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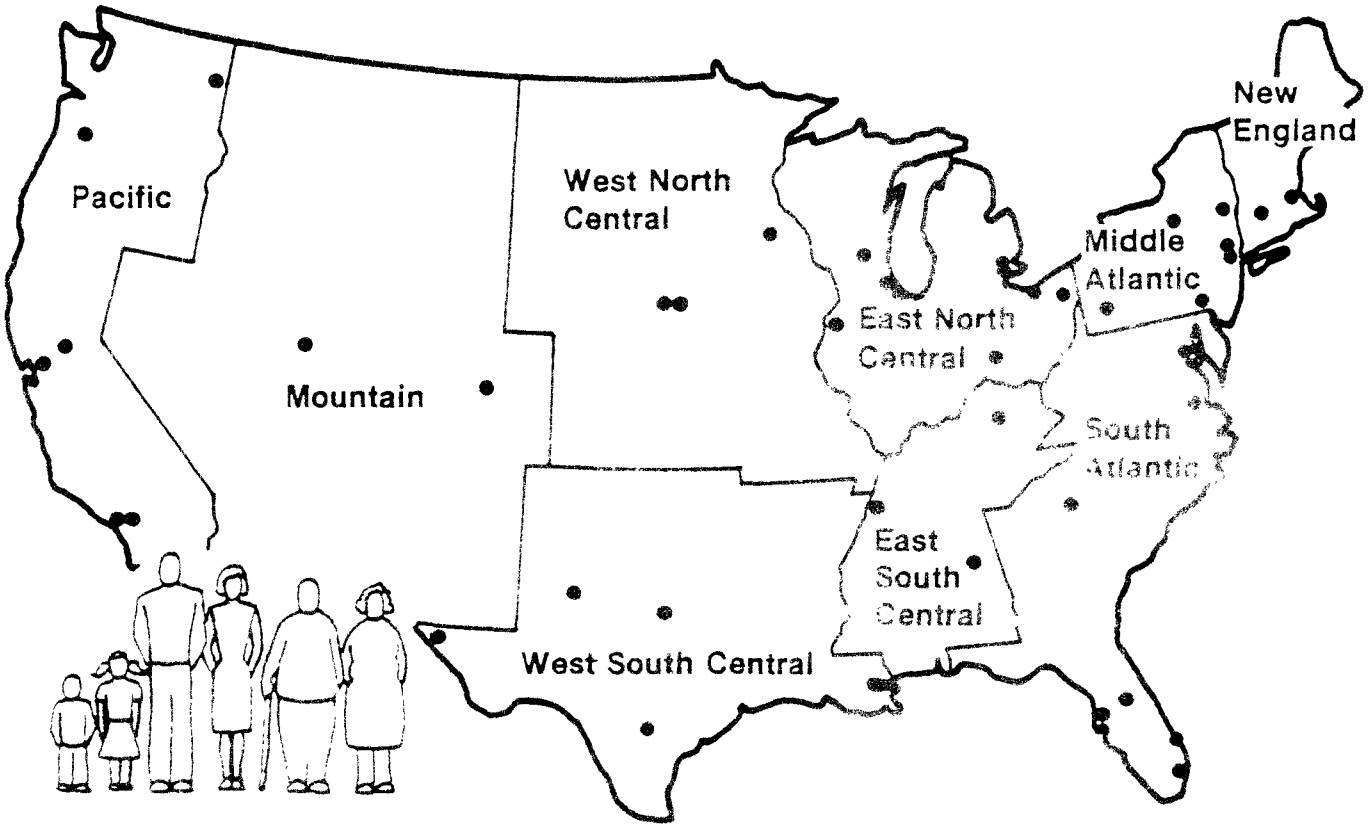
BROAD SCAN ANALYSIS OF THE FY82 NATIONAL HUMAN ADIPOSE TISSUE SURVEY SPECIMENS

VOLUME V - TRACE ELEMENTS



BROAD SCAN ANALYSIS OF HUMAN ADIPOSE TISSUE
VOLUME V: TRACE ELEMENTS

By

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FINAL REPORT

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DISCLAIMER

This document is a preliminary draft. It has not been released formally by the Office of Toxic Substances, Office of Pesticides and Toxic Substances, U.S. Environmental Protection Agency. It is being circulated for comments on its technical merit and policy implications.

PREFACE

This report is the final document of a five-volume series that details the broad scan chemical analysis of composite adipose tissue samples. These composite samples were prepared from individual specimens obtained from the Environmental Protection Agency's (EPA) National Human Adipose Tissue Survey (NHATS) fiscal year 1982 (FY82) repository.

This final volume summarizes data generated from the analysis of selected samples for trace elements using two multielement analysis techniques, inductively coupled plasma-atomic emission spectrometry (ICP-AES) and neutron activation analysis (NAA). Volume I, the Executive Summary, provides a synopsis of all analysis efforts completed under the broad scan analysis program. Volumes II through IV deal specifically with the chemical analysis of the NHATS composites for general volatile organics, semivolatile organics, and polychlorinated dibenzo-p-dioxins (PCDD) and dibenzofurans (PCDF). The statistical analyses of the data reported in these volumes will be reported separately by the EPA's Office of Toxic Substances (OTS) Design and Development Branch contractor, Battelle Columbus Laboratories.

The entire series of reports are references as follows:

- Stanley JS. 1986. Broad scan analysis of human adipose tissue: Volume I: Executive summary. EPA 560/5-86-035.
- Stanley JS. 1986. Broad scan analysis of human adipose tissue: Volume II: Volatile organic compounds. EPA 560/5-8-036.
- Stanley JS. 1986. Broad scan analysis of human adipose tissue: Volume III: Semivolatile organic compounds. EPA 560/5-86-037.
- Stanley JS. 1986. Broad scan analysis of human adipose tissue: Volume IV: Polychlorinated dibenzo-p-dioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs). EPA 560/5-86-038.
- Stanley JS, Stockton RA. 1986. Broad scan analysis of human adipose tissue: Volume V: Trace elements. EPA-560/5-86-039.

These method development, sample analyses, and reporting activities were completed for the EPA/OTS Field Studies Branch (FSB) broad scan analysis of human adipose tissue program (EPA Prime Contract Nos. 68-02-3938 and 68-02-4252, Work Assignments 8 and 21, respectively, Ms. Janet Remmers, Work Assignment Manager and Dr. Joseph Breem. Project Officer).

The experimental design for selecting samples for trace element analysis and preparing the composite samples from the NHATS repository for the broad scan analysis of organics was provided by Dr. Gregory Mack, Battelle Columbus Laboratories, under contract to the EPA/OTS Design and Development Branch (Mr. Phillip Robinson, Task Manager and Ms. Cindy Stroup, Program Manager).

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EXECUTIVE SUMMARY

The U. S. Environmental Protection Agency's Office of Toxic Substances (EPA/OTS) maintains a unique program for monitoring human exposure to potentially toxic substances through the National Human Adipose Tissue Survey (NHATS). NHATS is a statistically designed annual program to collect and analyze a nationwide sample of adipose tissue specimens for toxic compounds. NHATS focuses on documenting trends in human exposure to environmentally persistent contaminants, specifically, organochlorine pesticides and polychlorinated biphenyls (PCBs).

EPA/OTS has recognized a need to expand the use of the NHATS program to provide a more comprehensive assessment of toxic substances that accumulate in adipose tissues. The NHATS specimens collected during fiscal year 1982 (FY82) were designated for broad scan analysis. This broad scan analysis was used to detect volatile and semivolatile organic compounds and trace elements.

This report deals specifically with the measurement of trace elements in adipose tissue specimens from the FY82 NHATS repository. The objective of this study was to provide EPA/OTS with (1) a preliminary assessment of multielement analytical techniques that are applicable for determining trace elements in adipose tissues, and (2) provide a qualitative assessment of the level of the specific tissue elements that were present in selected specimens.

The analyses of nine selected adipose tissue specimens from the FY82 NHATS repository were completed using two multielement techniques: inductively coupled plasma-atomic emission spectroscopy (ICP-AES) and neutron activation analysis (NAA). A total of 18 elements were detected by the two techniques. The estimated tissue levels are reported.

Elements determined by ICP-AES were aluminum, boron, calcium, iron, magnesium, sodium, phosphorus, tin, and zinc. The estimated detection limits for 20 additional elements determined by ICP-AES are also reported. Elements determined by NAA were bromine, chlorine, cobalt, iron, iodine, potassium, sodium, rubidium, selenium, silver, and zinc. The estimated detection limits for 56 additional elements determined by NAA are also reported. The results reported for iron, zinc, and sodium are comparable for the two methods.

The results of this study are compared with tissue data reported in a monograph prepared for the International Commission in Radiological Protection (ICRP) (Snyder, Cook, Nasset, Karhausen, Howells, Tipton 1975). The data in the ICRP report are based on multiple sample analyses by single element techniques. The ICRP data were generated in the mid-1950s through the mid-1960s. The data for the FY82 NHATS specimens are generally comparable with the levels presented in the ICRP summary with the exception of tin. Tin was detected at concentration levels estimated to range from 4.6 to 15 μg/g in the NHATS specimens compared to 0.047 μg/g for the values in the ICRP report. These tin levels were estimated from the ICP-AES analyses but were not confirmed by NAA.

I. INTRODUCTION

The National Human Adipose Tissue Survey (NHATS) is the main operative program of the National Human Monitoring Program (NHMP). The NHMP was first established by the U.S. Public Health Service in 1967 and was subsequently transferred to U. S. Environmental Protection Agency (EPA) in 1970. During 1979 the program was transferred within EPA to the Exposure Evaluation Division of (EED) the Office of Toxic Substances (OTS).

NHATS is an annual program to collect a nationwide sample of adipose tissue specimens and to chemically analyze them for the presence of toxic compounds. The objective of the NHATS program is to detect and quantify the prevalences of toxic compounds in the general U.S. population. The NHATS data address part of OTS's mandate under TSCA to assess chemical risk to the U.S. population. The specimens are collected from autopsied cadavers and surgical patients according to a statistical survey design (Lucas, Pierson, Myers, Handy 1981). The survey design ensures that specified geographical regions and demographic categories are appropriately represented to permit valid and precise estimates of baseline levels, time trends, and comparisons across subpopulations.

The data for the NHATS are generated on an annual basis by collecting and chemically analyzing adipose tissue specimens for selected toxic substances. Historically, organochlorine pesticides and polychlorinated biphenyls (PCBs) have been the compound of interest.

A. Broad Scan Analysis Strategy

EPA/OTS recognized the need to provide a more comprehensive assessment of the toxic substances that accumulate in adipose tissue. An aggressive strategy to assess TSCA-related substances that persist in the adipose tissue of the general U.S. population has been developed by EED. The NHATS specimens collected during fiscal year 1982 (FY82) were selected for a broad scan analysis of volatile and semivolatile organic TSCA-related chemicals and trce elements (Mack, Stanley 1984).

B. Work Assignment Objectives

The objective of this phase of broad scan analysis work assignment was to determine the specific trace elements and the appropriate concentrations in human adipose tissue. The data generated from this study will serve as a preliminary basis for assessing the appropriateness of using human adipose tissue for monitoring exposure to potentially toxic trace elements.

This report deals specifically with the measurement of trace elements in selected human adipose tissue samples via two multielemental analysis techniques. Inductively coupled plasma-atomic emission spectrometry (ICP-AES) and neutron activation analysis (NAA). A computer-assisted literature review completed before this analysis task showed that there was very little information on the trace element composition of human adipose tissue. (Barry 1981; Bryne, Kosta 1978; Casey, Guthrie, Robinson 1982; Gross, Pfitzer, Yeager, Kehoe 1975; Kowal, Johnson, Kraemer, Pahren 1979; Mangelson, Hill, Nielson, Eatough, Christensen, Izatt, Richards 1982; Snyder, Cook, Nasset, Karhausen, Howells, Tipton 1975; Sumino, Hayakawa, Shibata, Kitamura 1975).

Hence, the results of this task produced valuable information on the levels of specific trace elements in human adipose tissue.

C. Organization of This Report

Section III of this volume describes the analytical procedures for the two multielement techniques. Section IV presents the results of the adipose tissue analyses. Section V summarizes the quality control (QC) procedures and analysis results. Section VI is a compilation of the pertinent references. Appendix A is included to provide examples of the emission responses observed for inductively coupled plasma-atomic emission spectroscopy (ICP-AES) wavelength scans characteristic for each element as measured for a blank, a calibration standard, and an actual sample.

II. RECOMMENDATIONS

The study reported in this document compares ICP-AES and neutron activation analysis (NAA) for multielement determination in adipose tissue. It is recommended that the sensitivity, selectivity, and cost of each analysis/technique be considered with respect to the trace elements of interest before proceeding with analysis of additional samples. The data collected from this preliminary scan of metals demonstrate that ICP-AES has sufficient sensitivity to allow analysis of large numbers of adipose samples for multiple elements at a reasonable cost. However, method modifications are necessary to lower the detection limits. These modifications include increasing the sample size and incorporation of an acceptable approach for correcting background resulting from overlapping spectral interferences.

NAA has the advantage of detecting some elements not possible by ICP-AES such as the halogens, rubidium, and cesium. Although multielement analysis by NAA results in considerably higher costs, it may provide the necessary sensitivity and specificity for elements of particular interest to EPA.

One other analytical technique that should be considered is high temperature graphite furnace atomic absorption spectrometry. This technique can provide lower levels of detection but is limited to single element measurements. This technique can be evaluated for elements of special interest.

A study of possible interest to EPA would be the determination of elements directly associated with the lipid materials, rather than the whole tissue. This would require a methods development effort since metal complexes will be affected by the acid digestion procedure described in the report. This could be accomplished by rendering the adipose tissue followed by multielement analysis of the oily materials. Based on the results of these studies, further evaluation may be necessary to determine speciation of specific elements.

A national survey of human adipose tissue to determine prevalence of toxic trace elements will require stringent quality assurance practices. This will require method validation for each element of interest, development of a representative reference material, and integration of a minimum quality

control program that specifies the frequency of analysis of blanks, spiked tissues, and reference materials. A representative reference material can be generated by isolating and homogenizing lipid materials from tissues collected through the NHATS program. Repetitive analysis of such a reference material (spiked and unspiked) can provide the necessary data to document method precision and accuracy for all samples analyzed.

III. EXPERIMENTAL

A. Human Adipose Tissue Samples

The adipose tissue samples were randomly selected from a set of specimens identified by the EPA Design and Development Branch contractor, Battelle Columbus Laboratories. Criteria for selecting the nine specific tissue samples included ample mass for two multielement techniques (greater than 10 g). The samples were visually checked to verify that the contents were primarily fatty tissues.

B. Instrumentation

1. Inductively Coupled Plasma-Atomic Emission Spectrometry

The adipose tissues were analyzed using a Jarrell-Ash Model 1155A inductively coupled argon emission spectrometer at MRI. The analytical operating conditions were:

Forward Power (kw): 1.15	Coolant Gas Flow (L/min): 18
Reflected Power (w): < 1.0	Auxiliary Gas Flow (L/min): 0
Observation Height (mm): 18	Sample Gas Flow (L/min): 0.4
Nebulizer: Fixed cross-flow	Solution Uptake (mL/min): 1.8
Peristaltic pump	

Operation and calibration procedures followed the manufacturer's guidelines. (Jarrell-Ash Division 1979) A calibration curve was generated using a 20% (v/v) nitric acid blank and a 10 µg/mL multielement standard. The multielement standard was prepared from commercially available 100 µg/mL stock (Spex Industries, Edison, New Jersey) in 20% (v/v) nitric acid.

2. Neutron Activation Analysis

The NAA was performed by General Activation Analysis, Inc., in San Diego, California using 4096 and 8192 channel gamma ray spectrometer systems equipped with Ge(Li) detectors after irradiation in a TRIGA® Mark 1 reactor. The very short-lived isotopes were determined 1 min after irradiation at a flux of 2.5×10^{12} n/cm^{2}s for 1 min. The short-, medium-, and long-lived isotopes were determined 1 h, 1 day, 1 wk, and 3 wk after irradiation for 30 min at a flux of 1.8×10^{12} n/cm^{2}s.

C. Sample Preparation

1. Inductively Coupled Plasma-Atomic Emission Spectrometry

Approximately 0.5-g aliquots of the adipose tissue specimens and associated quality control samples were placed in weighed glass culture tubes with Teflon®-lined screw-caps. Four milliliters of 50% (v/v) nitric acid that contained 9.96 µg of yttrium (Y) was added to each tube. The loosely capped tubes were allowed to stand at room temperature for 30 min and were the placed in an oven at 110°C for 1 h. The tubes were removed from the oven to tighten the caps and then were returned to the oven for 30 min. The digestion tubes were removed from the oven, the caps were loosened to relieve the pressure, and then were returned to the oven for an additional 30 min. The tubes were removed from the oven to cool and the samples were diluted with deionized water (greater than 18 megaohm resistivity) to a weight of approximately 10 g. The tubes were resealed and returned to the oven for 15 min. The tubes were removed from the oven, cooled, and the final digestate weights were measured and recorded.

2. Neutron Activation Analysis

Minimal sample preparation was required. The quantities of adipose tissue listed in Table 1 were sealed in polyethylene vials, irradiated in the TRIGA® Mark 1 reactor, and analyzed. Two different aliquots of each sample were irradiated at varying time spans (1-30 min) to determine the short- and long-lived isotopes.

D. Detection Limits

1. Inductively Coupled Plasma-Atomic Emission Spectrometry

Detection limits of the ICP-AES were determined by two criteria: (a) the mean and standard deviation concentrations were calculated for 10 replicate analyses of a quality control adipose sample. Three times the standard deviations were selected as the method limits of detection for the element. The adipose sample used for this analysis is identified as sample C-1 and is described with the ICP-AES method quality control procedures in this report. (b) The quality control adipose sample was scanned across a 2 Angstrom region of the spectrum to determine spectral interferences, which may result in false values. Interferences were observed for silver, arsenic, barium, beryllium, cadmium, cobalt, chromium, copper, mercury, potassium, manganese, molybdenum, nickel, lead, antimony, selenium, titanium, and thallium. The method limit of detection for each of these elements was calculated as the highest false positive bias plus three times the standard deviation determined from the 10 replicate analyses.

Appendix A contains copies of the wavelength scans for all elements. The x-axis is absolute emission counts. These scans are superimposes on each plot. These include (1) a calibration blank (VSTD1), (2) a standard equivalent to 10 µg/g ($AGHNO_3$, VSTD2, SSTD3 or SSTD4), and (3) the observed response for a quality control adipose sample (C-1).

Table 1. Mass of Adipose Tissue Analyzed by NAA

NHATS sample no.	Sample mass (grams)	
	Short irradiation[a]	Long irradiation[b]
8110967	0.6258	8.0992
8200586	1.0450	11.4472
8201022	0.6621	11.6512
8201428	0.7270	9.9623
8202046	0.7679	9.9502
8202962	0.9964	10.5311
8204083	0.8747	12.2480
8205874	0.9221	11.3542
8206278	1.0401	11.7879
NBS liver SRM 1577	0.2223	3.7243
NBS spinach SRM 1570	0.1989	2.8454

[a]Sample irradiated for 1 min for measurement of short-lived radioisotopes.
[b]Sample irradiated for 30 min for measurement of long-lived isotopes.

2. Neutron Activation Analysis

Method detection limits for the NAA were determined as six times the standard deviation of the noise level or the concentration required to generate a photopeak 10% greater than the baseline.

E. Quality Assurance

1. Inductively Coupled Plasma-Atomic Emission Spectrometry

The accuracy of the ICP analysis method was determined by preparation and analysis of spiked reagent blanks, NBS Spinach No. 1570, NBS Bovine Liver No. 1577a, and spiked NBS Bovine Liver 1577a. The spiked reagent blanks and spiked NBS Bovine Liver were prepared by adding approximately 50 µg of each element to the respective matrix.

Three subsamples (approximately 0.5 g each) of a bulk adipose sample were prepared by the procedures described for the NHATS specimens and were analyzed to evaluate the precision of the ICP-AES method. These subsamples were labeled C-1, C-2, and C-3. These subsamples are referred to as replicates of a single sample. However, no effort was exerted to homogenize the matrix before subsampling.

An instrument check standard (ICS2) was analyzed every 10th sample during the analysis of the adipose tissue to monitor the ICP-AES calibration. The metal concentration of the instrument check standard was equivalent to 0.5 µg/g for each of the elements.

Two complete method blanks were prepared following the procedures described for the NHATS specimens, although no adipose sample was added to the nitric acid digestion procedure. The responses observed for the reagent blanks were used for background contribution corrections with the actual samples.

An internal standard, yttrium, was added to each digested sample prior to the ICP-AES digestion procedure to monitor nebulization efficiency. The amount added was 9.96 µg per sample.

2. Neutron Activation Analysis

The accuracy of NAA was determined by preparation and analysis of NBS SRMs 1577 (bovine liver) and 1570 (spinach). Empty polyethylene vials were carried through the irradiation procedure and analysis to document and correct for background contribution.

IV. RESULTS

A. Inductively Coupled Plasma-Atomic Emission Spectrometry

The results of the ICP-AES analysis for the nine adipose tissues and the three quality control replicates (C-1, C-2, and C-3) are shown in Table 2. Only those elements that had concentrations above the estimated

detection limit for one or more of the samples are listed. Sodium and phosphorus were detected in all samples at concentrations of greater than 100 µg/g based on wet tissue weight. Calcium, iron, magnesium, tin, and zinc were also present in all samples. Aluminum was detected in 7 of the 12 samples. Boron was detected in only the three quality control bulk adipose tissue samples.

1. Precision

The triplicate analyses of a bulk adipose tissue sample (C-1, C-2, and C-3) demonstrated acceptable precision (Table 3) for all elements between samples except for aluminum and boron. Aluminum was detected in only one of the three replicates, while boron was detected in all three samples. The range of relative standard deviations for the analysis ranged from 13% for phosphorus to 57% for boron.

Table 3 also summarizes the precision of the 10 replicate ICP-AES analyses of sample C-1. These results demonstrate relative standard deviations ranging from 0.50% for magnesium up to 7.6% for aluminum. Since the detectable levels of aluminum are essentially a trace value (2.5 x the limit of detection), this difference in precision might reflect the ICP-AES performance near the detection limit. Based on the precision of the multiple analyses of sample C-1 (Table 3), the variability of the other elements could have been caused by sample preparation or a lack of homogeneity among the three subsamples.

2. Accuracy

Table 4 lists recoveries of the selected elements from fortified reagent blanks. These elements correspond to the analytes detected in the adipose tissue by ICP-AES. The spike recoveries for these elements ranged from 89.4 to 119%. The recovery of 119% was determined for iron using a secondary characteristic emission wavelength at 2714 Angstrom compared to 103% for the response observed at the primary wavelength at 2599 Angstroms. The values for iron reported in Table 2 were calculated using the primary characteristic wavelength at 2599 Angstroms.

Table 5 presents additional method accuracy data based on the recovery of the same six elements in spiked and unspiked NBS Bovine Liver 1577a. The accuracy for the ICP-AES analysis of the unspiked liver varied from 80.7% for iron to 114% for phosphorous compared to certified values. Again, it should be noted that the accuracy for iron varies from 80.7% at 2714 Angstroms compared to 90.1% at 2599 Angstroms. Concentration values for tin and boron were also determined for the bovine liver, although no certified value is available for comparison. The spike recovery values for the nine elements added to the certified reference material ranged from 87.2 to 128%. A recovery for phosphorous spiked to this sample was not reported since the spike level equivalent to 100 µg/g was negligible in comparison with the certified concentration of 11,100 µg/g.

Table 2. Concentration (μg/g) for Trace Elements in Adipose Tissue Determined by ICP-AES Analysis[a]

Element	NHATS FY82 sample number									Bulk adipose tissue[b]		
	8202046	8204083	8110967	8200586	8206278	8201428	8201022	8205874	8202962	C-1	C-2	C-3
Aluminum, Al	1.3	3.2	4.1	4.3	ND(0.63)[c]	8.3	ND(0.63)	ND(0.63)	2.1	1.6	ND(0.63)	ND(0.63)
Boron, B	ND(0.32)	ND(0.32)	ND(0.32)	ND(0.32)	ND(0.32)	ND(0.32)	ND(0.32)	ND(0.32)	ND(0.32)	22	13	6.3
Calcium, Ca	19	26	15	26	16	41	19	15	16	64	98	84
Iron, Fe[d]	13	26	8.3	7.6	3.0	12	7.3	36	20	14	22	18
Magnesium, Mg	10	11	8.8	25	6.5	12	8.4	14	17	10	15	14
Sodium, Na	540	640	280	380	240	430	150	240	270	230	280	320
Phosphorus, P	170	180	150	160	130	210	160	220	220	170	200	220
Tin, Sn	8.7	10	14	9.9	8.7	12	15	12	11	6.7	7.3	4.6
Zinc, Zn	1.6	2.6	1.6	3.3	1.1	1.8	3.2	6.0	4.1	1.4	1.8	1.9

[a] All values corrected for background contribution from the reagent blanks.
[b] A bulk adipose tissue specimen was analyzed as three replicates to provide information on method precision.
[c] ND = not detected. Value in parenthesis is the estimated limit of detection.
[d] Values reported for iron were detected at 2599 Angstroms.

Table 3. Relative Standard Deviation Between Adipose Samples C-1, C-2 and C-3

Element	RSD (%)[a]	RSD (%)[b]
Al	c	7.6
B	56	0.68
Ca	21	0.95
Fe (2714 Å)	32	6.0
Fe (2599 Å)	20	0.51
Mg	19	0.50
Na	16	1.3
P	13	1.0
Sn	23	2.3
Zn	17	2.3

[a] RSD % = $\frac{\text{Standard deviation}}{\text{Mean concentration}}$ x 100; n = 3 replicates (C-1, C-2, C-3).

[b] Relative deviation of 10 instrumental analyses of sample C-1.

[c] Not calculated because C-2 and C-3 were below the detection limit.

Table 4. Fortified Reagent Blank Recoveries for ICP-AES

Element	Amount of fortification (µg)	Mean observed[a] concentration (µg/g)	Recovery (%)
Al (3082 Å)	96.4	93.9 ± 1.8(s)	97.4
Al (2373 Å)	96.4	95.5 ± 1.7(s)	99.0
B	95.6	85.4 ± 2.3(s)	89.4
Ca	96.0	94.5 ± 2.6(s)	98.4
Fe (2714 Å)	95.2	114 ± 2(s)	119
Fe (2599 Å)	95.2	97.9 ± 1.4(s)	103
Mg	92.4	104 ± 2(s)	112
Na	92.0	99.8 ± 1.2(s)	108
P	96.0	90.1 ± 1.8(s)	93.8
Sn	96.0	103 ± 1(s)	108
Zn	96.0	108 ± 1(s)	112

[a]Observed concentrations are blank corrected; (s) = standard deviation; n = 4.

Table 5. NBS Bovine Liver No. 1577A Recoveries for ICP-AES

Element	(Unfortified)[a] observed concentration (µg/g)	Known[b] concentration (µg/g)	Recovery (%)	Fortified[c] observed concentration (µg/g)	Fortification level (µg/g)	Recovery (%)
Al (3082 Å)	< 0.634	(2)	-	173	189	91.5
Al (2373 Å)	< 0.781	(2)	-	174	189	92.1
B	9.82 ± 1.67	d	-	175	187	88.3
Ca	130 ± 5	120	108	318	188	100
Fe (2714 Å)	156 ± 3	194	80.7	395	187	128
Fe (2599 Å)	182 ± 2	194	93.6	380	187	106
Mg	612 ± 15	600	102	832	181	122
Na	2,190 ± 26	2,430	90.1	2,347	180	87.2
P	12,641 ± 134	11,100	114	13,366	188	e
Sn	11.2 ± 2.7	d	-	181	188	90.3
Zn	115 ± 3	123	93.5	315	188	106

[a]Blank corrected; n = 4.
[b]Values in parentheses are not certified (information only).
[c]Blank corrected; n = 2.
[d]Element not certified by NBS.
[e]Fortification level too low for accurate recovery calculations.

3. Detection Limits

The detection limits for the ICP-AES technique presented in Table 6 are based upon a conservative means of calculation because instrumental background correction was not used. The detection limits are sufficiently high to overcome any false bias caused by spectral overlap or background shift. The incorporation of background correction could lower the detection limits considerably. In most cases, the background and spectral overlap corrected detection limits would be in the parts per billion (ng/g) to parts per million (μg/g) range.

B. Neutron Activation Analysis

Table 7 presents the elements detected by NAA at concentrations greater than detection limits for each sample. The elements determined by NAA were bromine, chlorine, cobalt, iron, potassium, sodium, and zinc. These elements were detected in all nine of the adipose tissue samples. Selenium and rubidium were detected frequently but not in all specimens. Gold, silver, and iodine were detected in only one or two specimens.

1. Accuracy

NBS standard reference materials (bovine liver SRM 1577a and spinach leaves SRM 1570) were analyzed with the nine adipose tissue samples to determine the accuracy of NAA. The results of the bovine liver analysis and the percent accuracy results are tabulated in Table 8. The NAA method shows good accuracy for all elements with reported certified values. The observed concentration for cobalt was determined to be 360% greater than the NBS value. As noted in Table 8, the value reported for cobalt by NBS is not certified. Although, since the data was provided from a nonreference method or was not determined by at least two independent methods. The results of the spinach leaves (SRM 1570) analysis and percent accuracy are tabulated in Table 9. Again, these results demonstrate good accuracy (91 to 100%) for all elements except chromium, zinc, thorium, and bromine, which range from 67 to 80% of the NBS reported values.

2. Detection Limits

The detection limits (Table 10) as calculated by General Activation Analysis, Inc., depend upon the amount of time between irradiation and analysis and the number and amount of interfering elements. Table 9 provides the detection limits for an additional 56 elements that were determined by NAA.

3. NAA Versus ICP-AES Results

The results of the NAA analyses for sodium, iron, and zinc are in agreement with the values reported for the corresponding samples analyzed by ICP-AES. Table 11 summarizes the data for these three elements as determined by the two multielement techniques. Differences in the values from the two analytical procedures might be attributed to inhomogeneity of the adipose tissue specimens. Concern for introducing contamination deterred any attempt to homogenize the specimens.

Table 6. ICP-AES Method Detection Limits for Human Adipose Tissue

Element	Detection limit (μg/g)[a]
Ag	3.1[b]
Al (3082 Å)	0.63
Al (2373 Å)	0.78
As	28[b]
B	0.32[b]
Ba	0.28[b]
Be	0.048
Ca	2.4
Cd	0.073[b]
Co	0.94[b]
Cr	2.3[b]
Cu	1.4[b]
Fe (2714 Å)	3.2
Fe (2599 Å)	0.28
Hg	5.9[b]
K	34[b]
Mg	0.22
Mn	0.12[b]
Mo	1.4[b]
Na	8.3
Ni	0.71[b]
P	6.6
Pb	6.1[b]
Sb	3.8[b]
Se	12[b]
Sn	0.36
Ti	0.36[b]
Tl	12[b]
Y	0.22
Zn	0.13

[a]The detection limit for a particular element was calculated as three times the standard deviation determined from 10 replicate analyses of the QC sample (C-1).

[b]The detection limit calculation indicates the consideration for interferences as determined from the QC sample (C-1).

Table 7. Summary of Data for Trace Elements (μg/g) Identified in Nine Human Adipose Tissue Specimens by NAA

Element	Sample number								
	8206278	8202962	8201022	8201428	8202046	8110967	8205874	8204083	8200586
Gold, Au	ND(0.0012)[a]	ND(0.0042)	ND(0.0017)	ND(0.0032)	ND(0.0025)	ND(0.0025)	ND(0.0018)	0.0030	0.0006
Bromine, Br	1.6	0.33	0.74	2.4	0.62	1.0	0.99	0.94	0.80
Chlorine, Cl	380	410	240	480	550	360	370	1,500	580
Cobalt, Co	0.034	0.073	0.035	0.079	0.078	0.065	0.047	0.055	0.044
Iron, Fe	3.5	26	6.3	14	9.6	6.3	40	7.7	9.3
Iodine, I	ND(1.5)	ND(2.5)	ND(1.4)	ND(3.4)	ND(1.9)	ND(6.0)	ND(3.1)	ND(8.6)	13
Potassium, K	52	200	180	110	95	90	270	250	200
Sodium, Na	290	340	240	410	490	310	320	1,200	580
Rubidium, Rb	0.21	0.27	0.23	0.24	0.18	ND(0.73)	ND(0.61)	ND(1.1)	0.25
Selenium, Se	0.034	ND(0.11)	0.038	0.056	0.030	ND(0.082)	0.042	0.033	0.042
Silver, Ag	ND(0.14)	ND(0.12)	ND(0.13)	ND(0.16)	ND(0.16)	0.38	ND(0.13)	ND(0.12)	ND(0.13)
Zinc, Zn	1.4	2.3	1.6	1.4	1.5	1.4	3.4	2.0	4.5

[a]ND = not detected. Value in parentheses is the estimated limit of detection.

Table 8. Results of the Neutron Activation Analysis (NAA) for Trace Elements in the NBS SRM 1577-Bovine Liver

Element	NBS certified[a] concentration	Observed concentration	Percent (%) accuracy
Potassium, K	0.97%	0.96%	99
Iron, Fe	270 ± 20 μg/g	260 μg/g	96
Zinc, Zn	130 ± 10 μg/g	140 μg/g	110
Rubidium, Rb	18.3 ± 1.0 μg/g	18 μg/g	98
Manganese, Mn	10.3 ± 1.0 μg/g	9.6 μg/g	93
Selenium, Se	1.1 ± 0.1 μg/g	1.1 μg/g	100
Chlorine, Cl	(2,600 μg/g)	2,700 μg/g	104
Cobalt, Co	(0.18 μg/g)	0.62 μg/g	360
Molybdenum, Mo	(3.2 μg/g)	3.4 μg/g	106

[a]Values in parentheses are not certified by NBS (information only). These elements are not certified because data were provided from nonreference methods, or were not determined by at least two independent methods.

Table 9. Results of the Neutron Activation Analysis (NAA) for Trace Elements in the NBS SRM 1570-Spinach

Element	NBS certified[a] concentration	Observed concentration	Percent (%) accuracy
Potassium, K	3.56 ± 0.03%	3.2%	90
Iron, Fe	550 ± 20 μg/g	530 μg/g	96
Manganese, Mn	165 ± 6 μg/g	155 μg/g	94
Zinc, Zn	50 ± 2 μg/g	40 μg/g	80
Rubidium, Rb	12.1 ± 0.2 μg/g	11 μg/g	91
Chromium, Cr	4.6 ± 0.3 μg/g	3.4 μg/g	74
Thorium, Th	0.12 ± 0.03 μg/g	0.08 μg/g	67
Bromine, Br	(54 μg/g)	42 μg/g	78
Cobalt, Co	(1.5 μg/g)	1.5 μg/g	100
Scandium, Sc	(0.16 μg/g)	0.15 μg/g	94

[a]Values in parentheses are not certified by NBS (information only). These elements are not certified because data were provided from nonreference methods, or were not determined by at least two independent methods.

Table 10. Calculated Detection Limits for Trace Elements from the Neutron Activation Analysis (NAA) of Nine Adipose Tissue Specimens

Element	Range of calculated detection limits (μg/g)[a]
Aluminum, Al	2.9-6.7
Arsenic, As	0.084-1.9
Barium, Ba	1.5-6.2
Calcium, Ca	110-460
Cadmium, Cd	2.7-7.1
Cerium, Ce	0.032-0.12
Chromium, Cr	0.076-0.21
Cesium, Cs	0.010-0.025
Copper, Cu	10-39
Dysprosium, Dy	0.43-0.97
Erbium, Er	3.0-14
Europium, Eu	0.0081-0.027
Fluorine, F	180-1,100
Gallium, Ga	1.8-5.9
Gadolinium, Gd	9.3-26
Germanium, Ge	58-130
Hafnium, Hf	0.0071-0.014
Mercury, Hg	0.0001-0.043
Holmium, Ho	0.031-0.072
Indium, In	0.035-0.16
Iridium, Ir	0.00010-0.00030
Lanthanum, La	0.060-0.17
Lutetium, Lu	0.0017-0.011
Magnesium, Mg	49-280
Manganese, Mn	0.23-0.54
Molybdenum, Mo	0.75-1,450
Niobium, Nb	62-340
Neodymium, Nd	0.15-0.43
Nickel, Ni	2.8-6.2
Osmium, Os	0.011-0.046
Phosphorus, P	1,900-4,400
Palladium, Pd	1.5-4.4
Praseodymium, Pr	8.1-25
Platinum, Pt	0.31-0.48
Rhenium, Re	0.0079-0.027
Rhodium, Rh	0.083-0.51
Ruthenium, Ru	0.032-0.076
Sulfur, S	3,900-17,000
Antimony, Sb	0.013-0.021
Scandium, Sc	0.0009-0.0050
Silicon, Si	420-960
Samarium, Sm	0.0021-0.0054

Table 10 (concluded)

Element	Range of calculated detection limits (μg/g)[a]
Strontium, Sr	17-28
Tantalum, Ta	0.010-0.026
Terbium, Tb	0.0028-0.0078
Tellurium, Te	0.21-2.0
Thorium, Th	0.0035-0.011
Tin, Sn	1.7-4.4
Titanium, Ti	7.6-42
Thallium, Tl	110-350
Thulium, Tm	0.0065-0.010
Uranium, U	0.022-0.045
Vanadium, V	0.062-0.25
Tungsten, W	0.22-0.56
Yttrium, Y	1,400-4,400
Ytterbium, Yb	0.0093-0.017
Zirconium, Zr	82-140

[a]Detection levels determined as six times the standard deviation of the observed noise level.

Table 11. Comparison of ICP-AES and NAA Results for Sodium (Na), Iron (Fe), and Zinc (Zn) in Selected Human Adipose Tissue Samples

NHATS specimen no.	Sodium (µg/g)			Iron (µg/g)			Zinc (µg/g)		
	ICP-AES	NAA	RPD[a]	ICP-AES	NAA	RPD	ICP-AES	NAA	RPD
8206278	240	290	19	3.0	3.5	15	1.1	1.4	24
8202962	270	340	23	20	26	26	4.1	2.3	56
8201022	150	240	46	7.3	6.3	15	3.2	1.6	67
8201428	430	410	4.7	12	14	15	1.8	1.4	25
8202046	540	490	9.7	13	9.6	30	1.6	1.5	6.5
8110967	280	310	10	8.3	6.3	27	1.6	1.4	13
8205874	240	320	29	36	40	11	6.0	3.4	55
8204083	640	1,200	61	26	7.7	110	2.6	2.0	26
8200586	380	580	42	7.6	9.3	20	3.3	4.5	31

[a]RPD = relative percent difference. Calculated as difference of the two values divided by the average of the two values and multiplied by 100.

Both analytical techniques are capable of determining the presence of tin at low µg/g concentrations. Tin was detected in the NHATS specimen by the ICP-AES method at concentrations ranging from 8.7 to 15 µg/g. However, these concentrations could not be confirmed by NAA with reported detection limits ranging from 1.7 to 4.4 µg/g for aliquots of the same adipose tissue specimens. Clarification of this discrepancy for this element will require additional effort. No major tin contribution was detected in the method reagent blanks, and it was anticipated that the NAA method detection limit may require verification through low level spikes.

C. Discussion

As noted in the introduction, very little information is available in the literature (Snyder et al. 1975; Sumino et al. 1975; Gross et al. 1975; Bryne, Kosta 1978; Kowal et al. 1979; Barry 1981; Casey et al. 1982; Mangelson et al. 1982) regarding the levels of specific elements in human adipose tissue. The most significant source of information was found in a report prepared for the International Commission on Radiological Protection (ICRP). (Snyder et al. 1975) This report summarizes elemental composition based on total body organ and tissue type for what is referred to as "reference man." The data presented in that report was taken from several literature sources, much of which is based on activities completed at the Oak Ridge National Laboratory and University of Tennessee from the mid 1950s to the mid 1960s. The report does not specify the analytical procedures used to obtain the data, although some general references are made to colorimetric, atomic emission, atomic absorption, and DC-Arc plasma emission techniques.

Table 12 compares the range of concentrations observed for specific elements from the NHATS specimens in this study to the estimates presented for "reference man" in the ICRP report (Snyder et al. 1975). The ICRP report specifies that "reference man" consists of a total mass of 70 kg (∿ 150 lb) with as much as 21% or 15 kg being adipose tissue. The general term, adipose tissue, in the ICRP report includes subcutaneous adipose, adipose surrounding organs such as the kidneys or intestines, and interstitial adipose interspersed among the cells of an organ and yellow marrow. In general, the data generated by the two multielement techniques are close to the information presented for "reference man." The most obvious differences are noted for boron, silver, and tin.

VI. QUALITY ASSURANCE/QUALITY CONTROL

As discussed in Section III of this volume, several samples were included to provide estimates of method accuracy and precisions through the analysis of spikes, replicates, and standard reference materials. The results of these analyses have been presented in Tables 3, 5, 6, 10, and 11. These data indicate that the ICP-AES and NAA multielement techniques can accurately and precisely measure trace elements in human adipose tissues.

Table 12. Comparison of Elements Selected in the NHATS FY82 Specimens and the ICRP Reference Man[a]

Element	Reported concentration (μg/g) NHATS FY82 specimens	ICRP reference man
Aluminum (Al)	ND(0.63)-4.3	0.35
Boron (B)	ND(0.32)-22	0.073
Bromine (Br)	0.33-2.4[b]	0.43
Calcium (Ca)	15-98	23
Chlorine (Cl)	360-1,500[b]	1,200
Cobalt (Co)	0.034-0.079[b]	0.024
Gold (Au)	ND-0.0030[b]	< 0.33
Iodine (I)	ND(1.4)-13[b]	c
Iron (Fe)	3.0-36 3.5-26[b]	2.4
Magnesium (Mg)	6.5-25	20
Phosphorus (P)	130-220	160
Potassium (K)	52-270[b]	320
Rubidium (Rb)	ND-0.27[b]	c
Selenium (Se)	ND-0.056[b]	c
Silver (Ag)	ND-0.38	0.0013
Sodium (Na)	150-540 240-1,200[b]	510
Tin (Sn)	4.6-15	0.047
Zinc (Zn)	1.1-6.0 1.4-4.5[b]	1.8

[a]Snyder WS, Cook MJ, Nasset ES, Karhausen LR, Howells GP, Tipton IH. 1975. Report of the task group on reference man. ICRP No. 23, [b]Pergamon Press, pp. 273-334.
[b]Values from NAA. All other values for the NHATS specimens were [c]observed by ICP-AES.
[c]No estimate provided.

In addition to these quality control checks, additional procedures monitored the performance of the ICP-AES technique. These quality control checks included routine analysis of a check standard to monitor calibration and the inclusion of an internal standard to monitor nebulization efficiency.

An instrumental check standard (ICS2) equivalent to 0.5 µg/g of each element was analyzed every 10th sample with the adipose tissue. Table 13 presents the results of the calibration check completed after the analysis of the NHATS specimens. These results demonstrate that the calibration standard falls outside the ± 5% control limit established from 10 replicates of the same standard analyzed previous to the adipose tissues. Since this calibration check is outside the control limits, the data should be classified as estimates.

The recovery of the internal standard, yttrium (Y), from all adipose tissue samples is presented in Table 14. As noted, the recoveries ranging from 97 to 112% indicate that no losses were experienced due to the efficiency of nebulization.

The objective of this study was to provide EPA/OTS with qualitative information on trace elements present in adipose tissue. Further exposure studies involving trace element measurements of adipose tissue should require a stringent protocol to maintain the control limits. Further studies normalize reported concentrations for water content of the tissues. This can be accomplished either through ashing or lyophilization, although the analyst must be aware that the more volatile elements may undergo losses with these techniques.

Table 13. ICP-AES Analysis of ICS2 Standard

Element	Control limit[a] (μg/g)	Measured value (μg/g)
Al (3082 Å)	0.530-0.586	0.686
Al (2373 Å)	0.529-0.585	0.744
As	0.502-0.554	0.750
B	0.501-0.553	0.509
Ba	0.431-0.476	0.421
Be	0.458-0.506	0.471
Ca	0.454-0.502	0.432
Cd	0.484-0.536	0.520
Co	0.455-0.503	0.504
Cr	0.491-0.543	0.572
Cu	0.464-0.513	0.482
Fe (2714 Å)	0.943-1.04	1.85
Fe (2599 Å)	0.451-0.499	0.460
Hg	0.518-0.572	0.635
Mg	0.468-0.518	0.474
Mn	0.444-0.490	0.455
Mo	0.407-0.449	0.468
Na	0.473-0.523	0.529
Ni	0.464-0.513	0.517
P	0.437-0.483	0.742
Pb	0.514-0.568	0.746
Sb	0.489-0.541	0.597
Se	0.501-0.553	0.731
Sn	0.476-0.526	0.608
Ti	0.426-0.470	0.426
Tl	0.494-0.546	0.646
Y	0.433-0.479	0.433
Zn	0.479-0.529	0.536

[a]The recommended control limits are ± 5% of the mean concentration of 10 replicate analyses.

Table 14. Y-Internal Standard Recovery

Sample	Y-% recovery[a]
8201022	104
8201428	100
8200586	106
8204083	97
8202962	101
8206278	103
8205874	102
8202046	98
8110967	98
C-1	105
C-2	110
C-3	112

[a]9.96 µg Y added to all samples.

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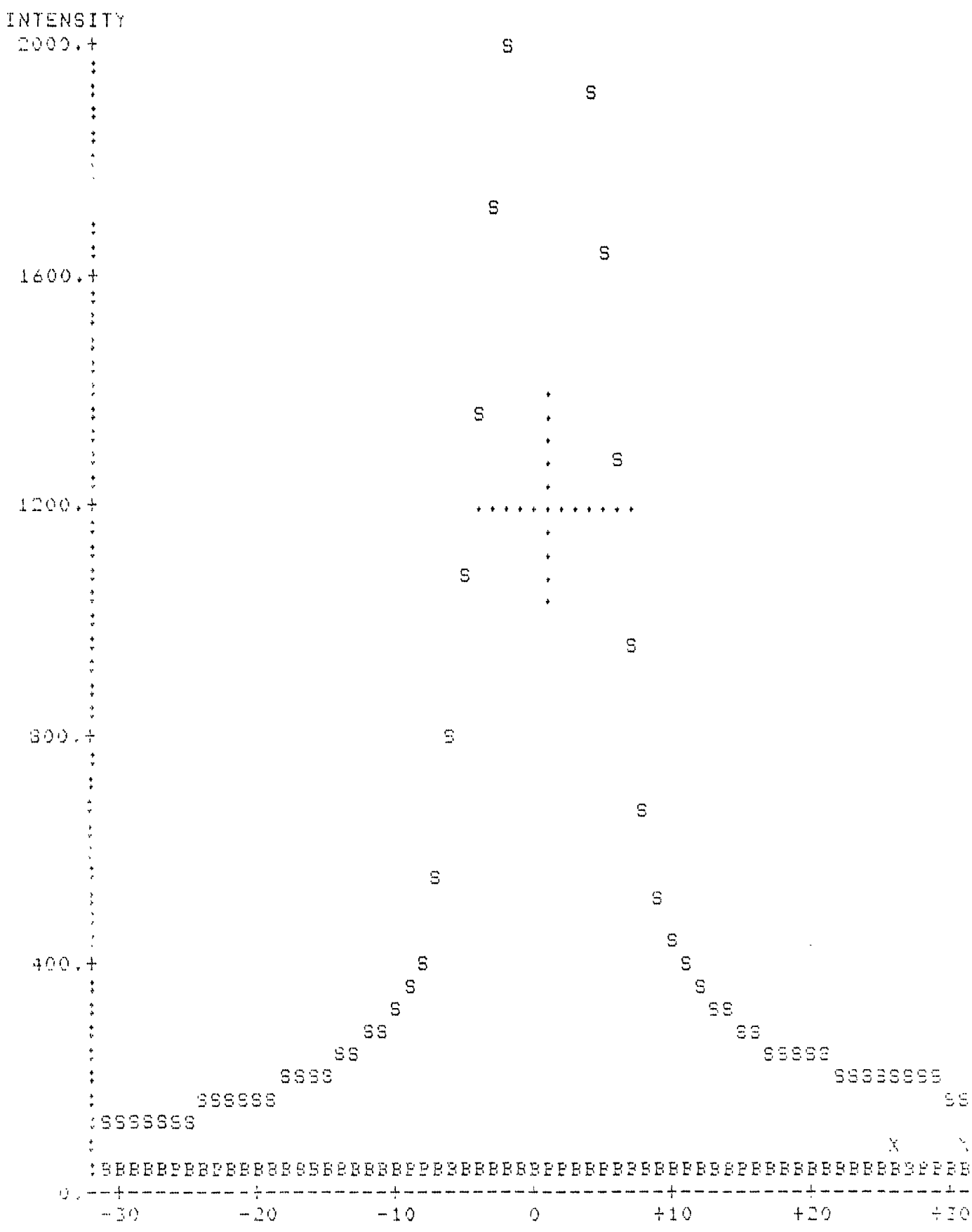
APPENDIX A

CHARACTERISTIC WAVELENGTH SCANS FOR INDIVIDUAL TRACE ELEMENTS IN BLANKS (VSTD1), 10 µg/g STANDARD (AGHNO3, SSTD3, SSTD4), AND ADIPOSE TISSUE (FAT, SAMPLE C-1)

WAVELENGTH SCANS FOR AG AT 3280 ANGSTROMS



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WAVELENGTH SCANS FOR AG AT 3280 ANGSTROMS

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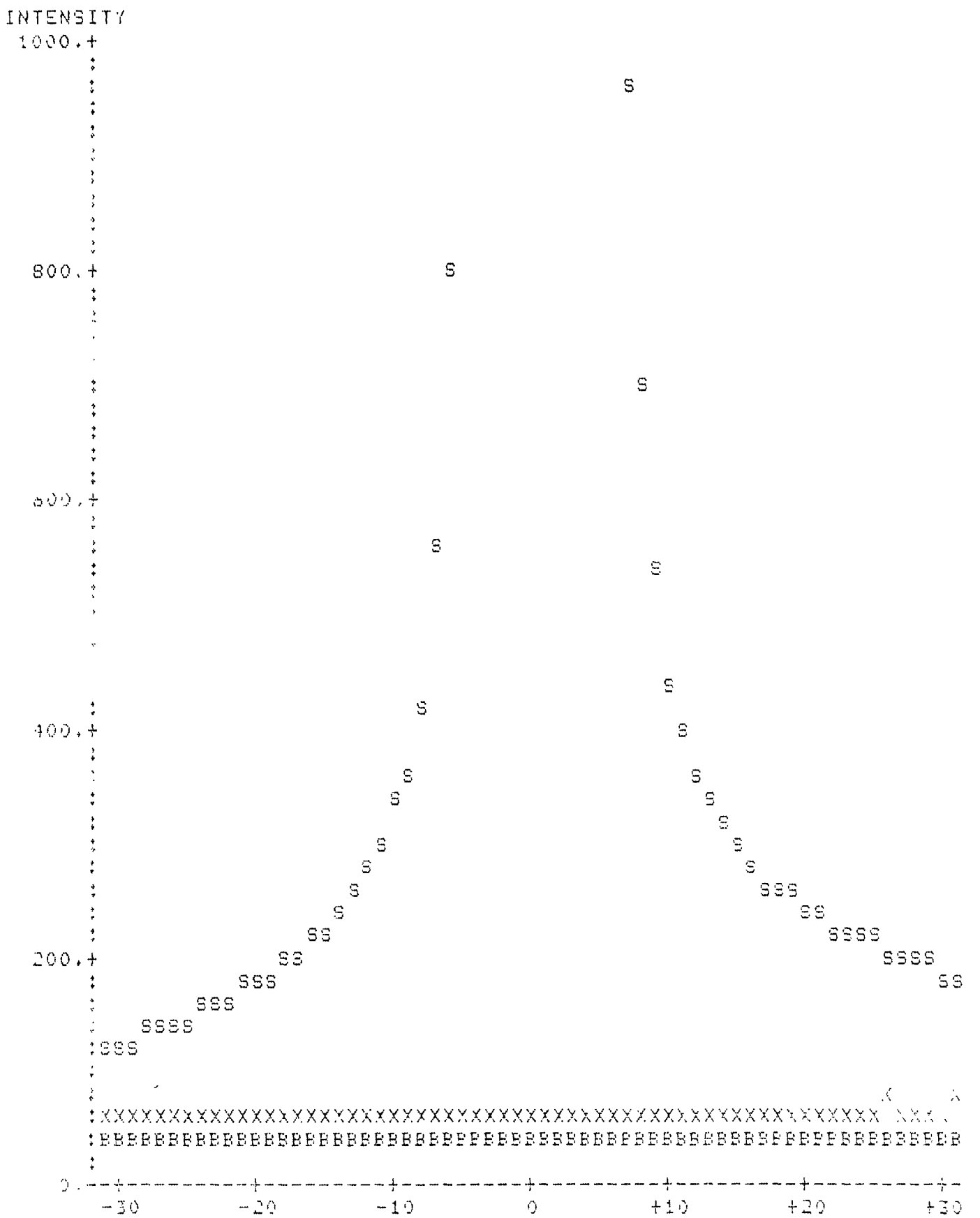

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WAVELENGTH SCANS FOR AL AT 3082 ANGSTROMS

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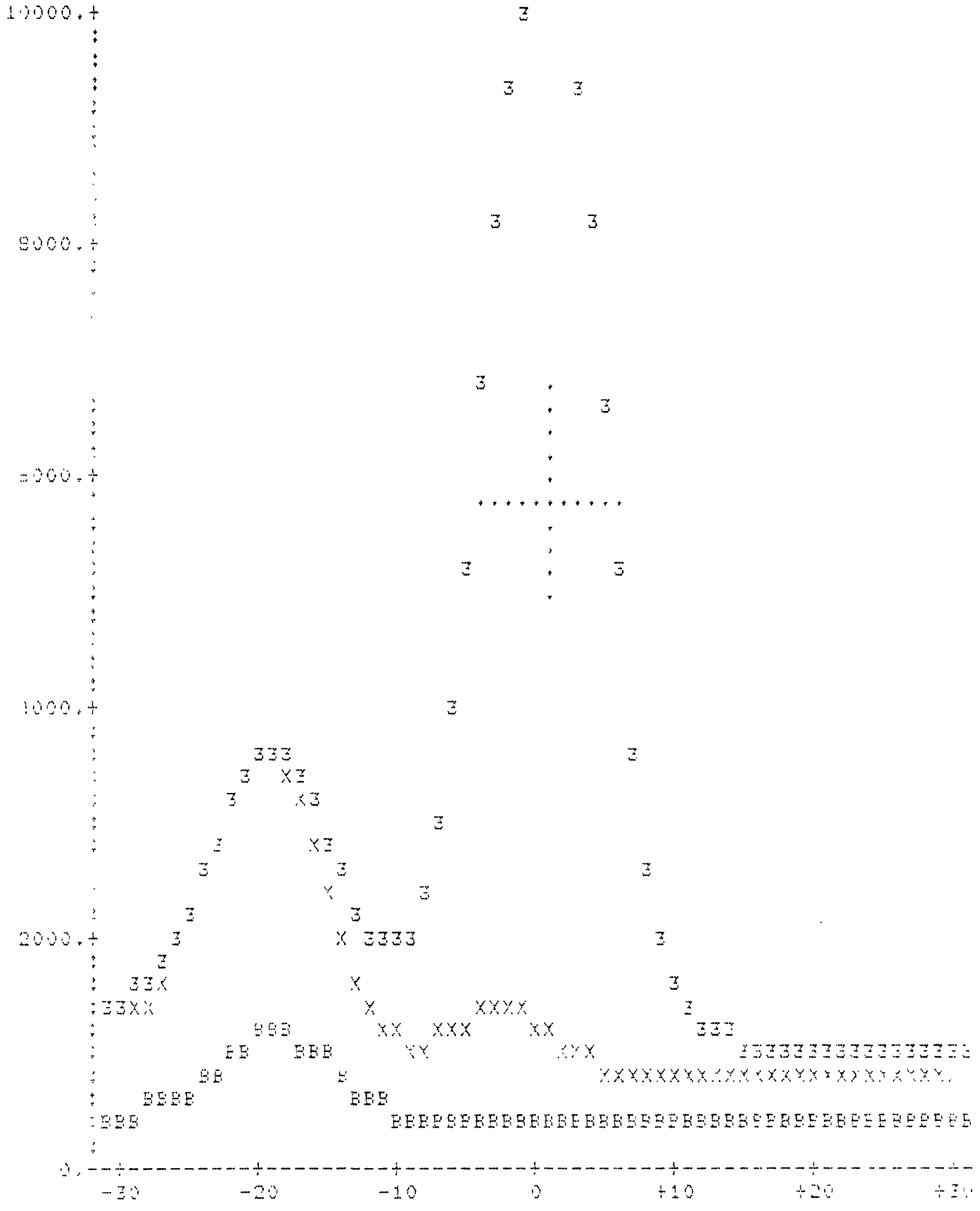

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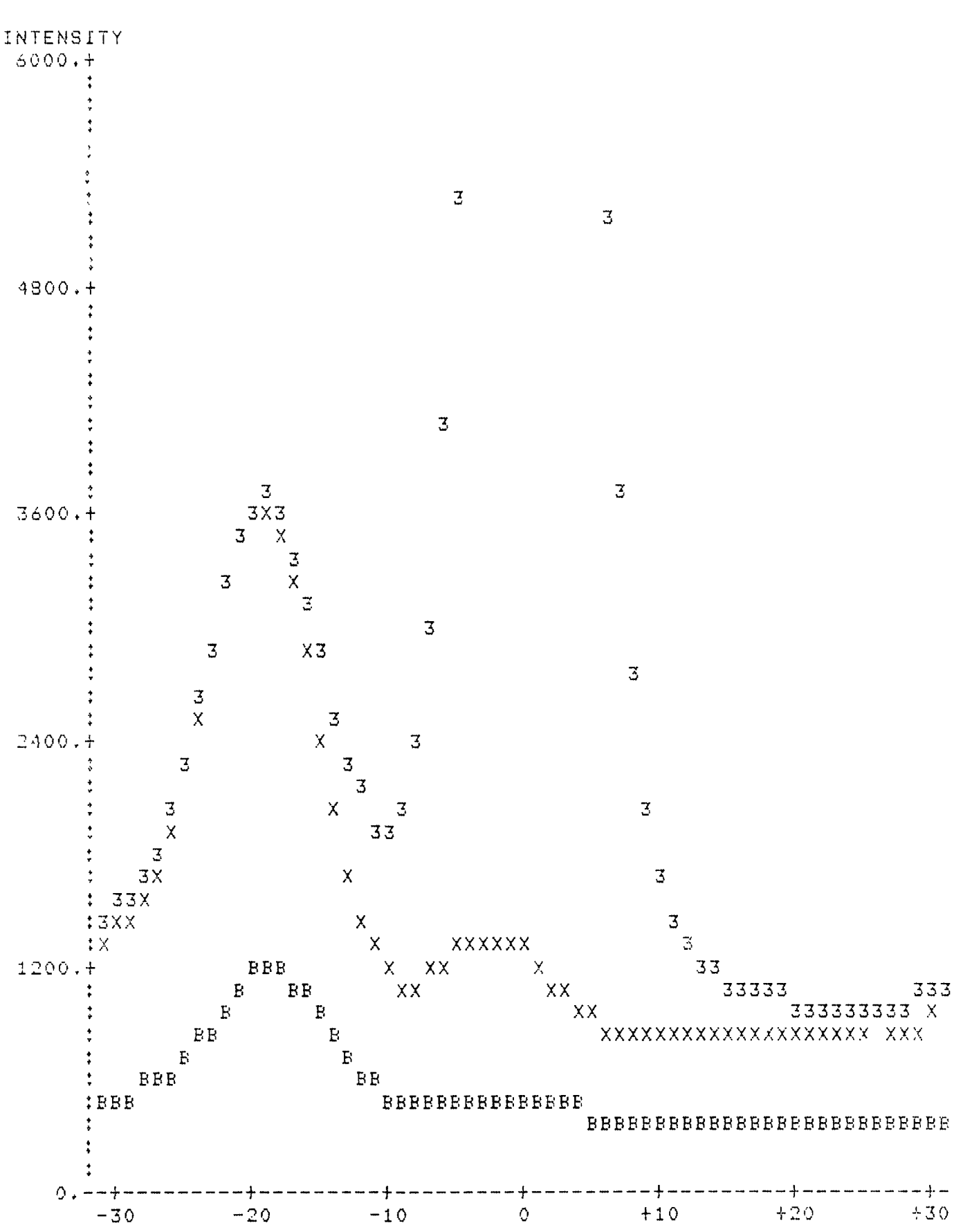

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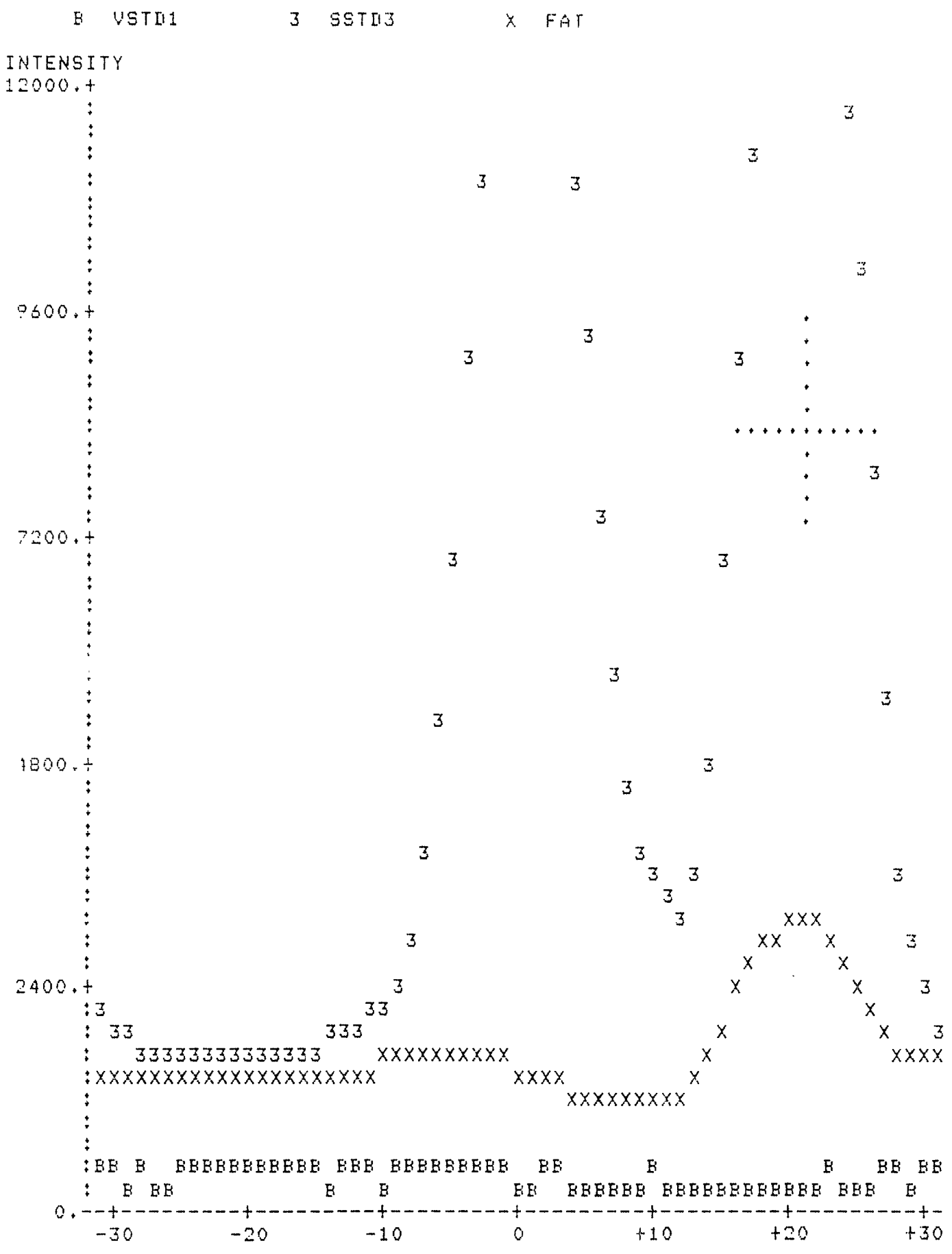

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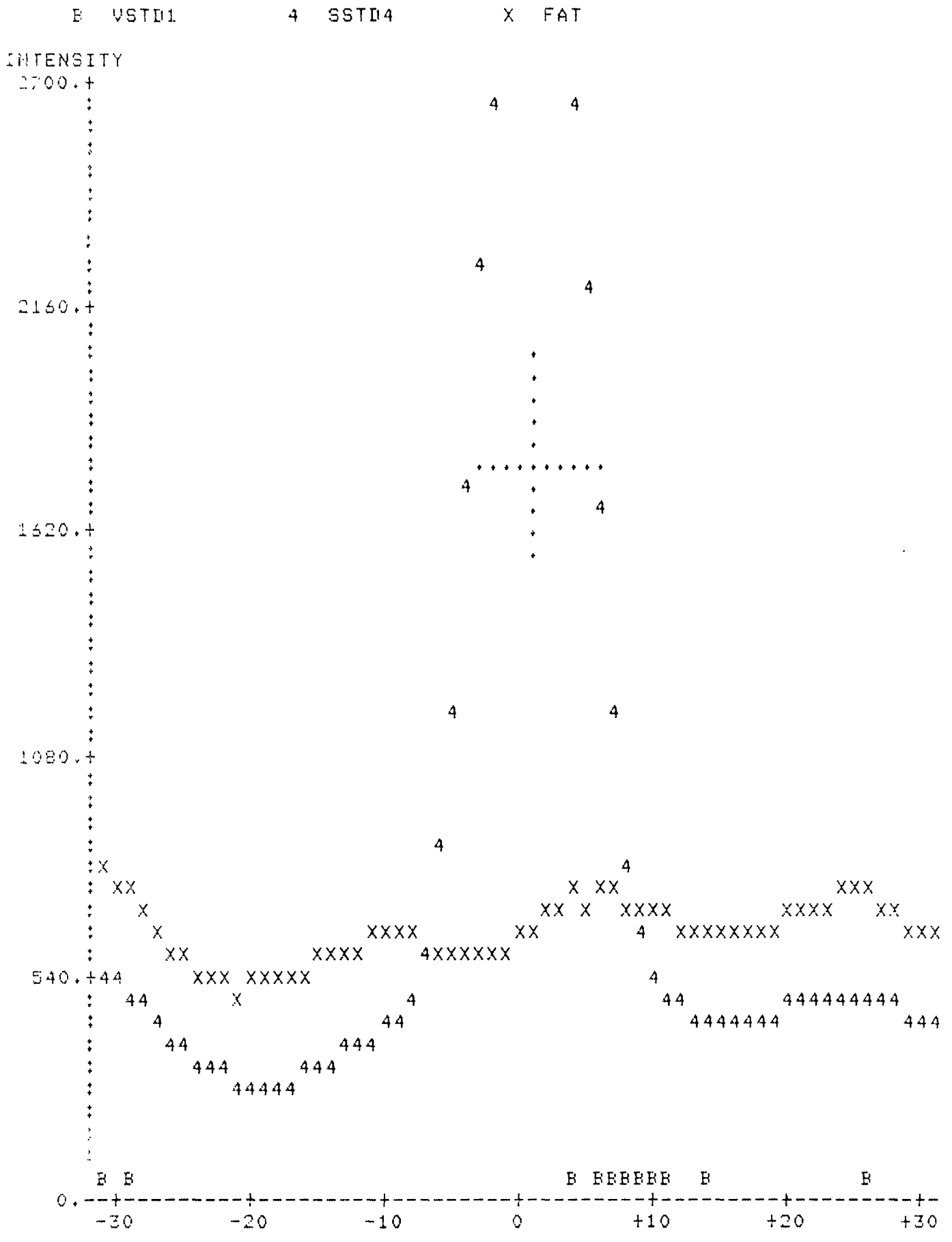
WAVELENGTH SCANS FOR AS AT 1936 ANGSTROMS



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WAVELENGTH SCANS FOR AS AT 1936 ANGSTROMS

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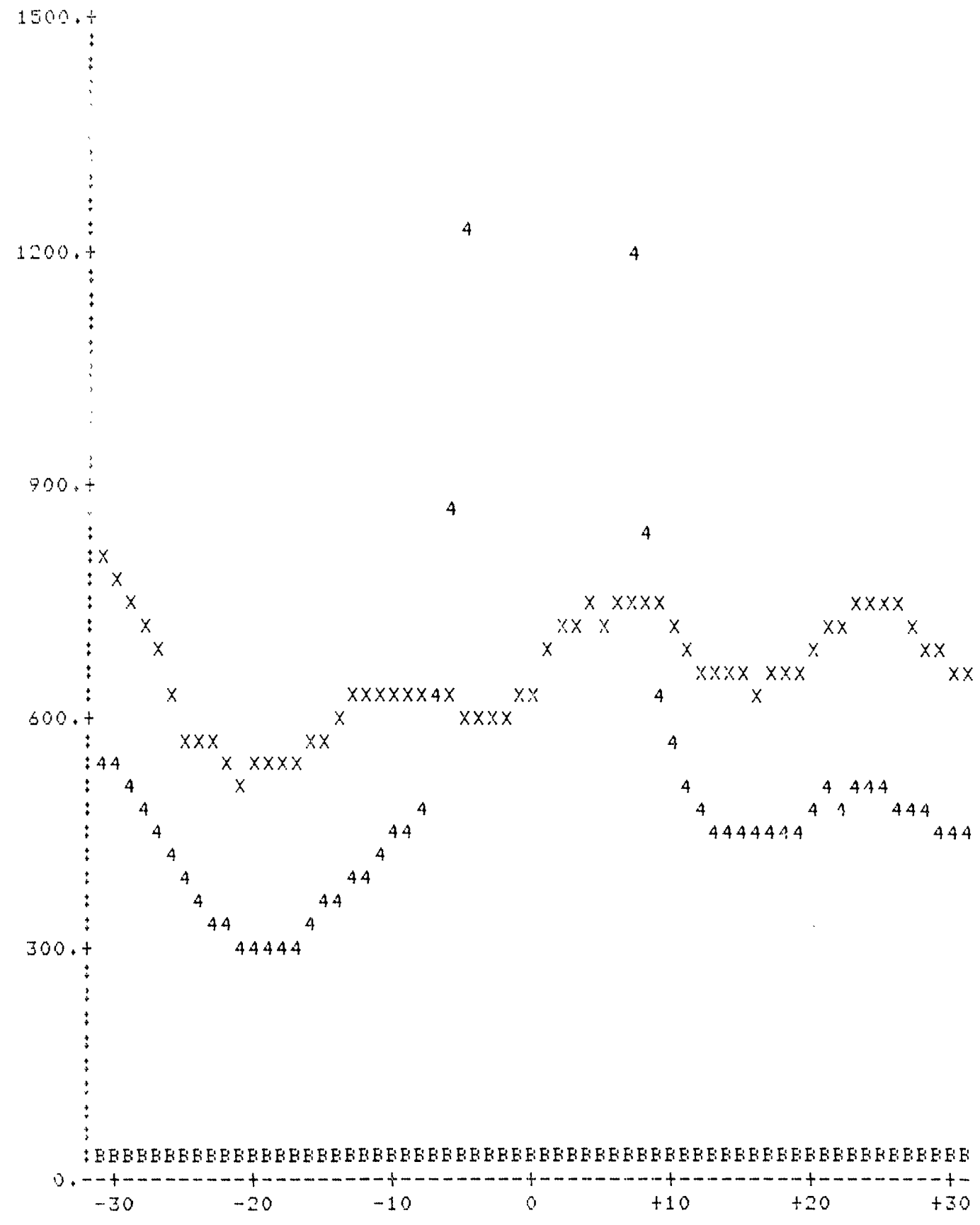

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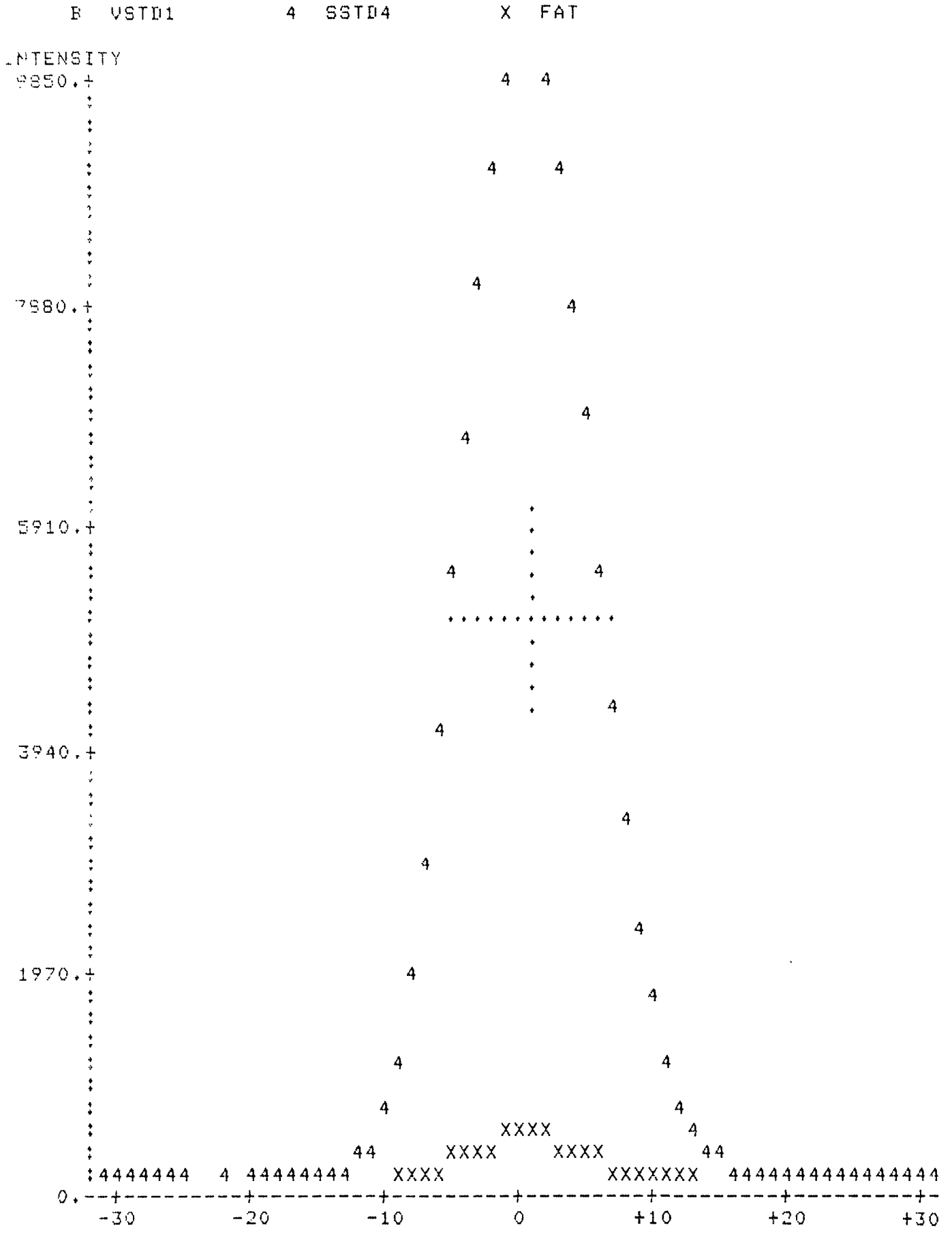

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WAVELENGTH SCANS FOR B AT 2496 ANGSTROMS

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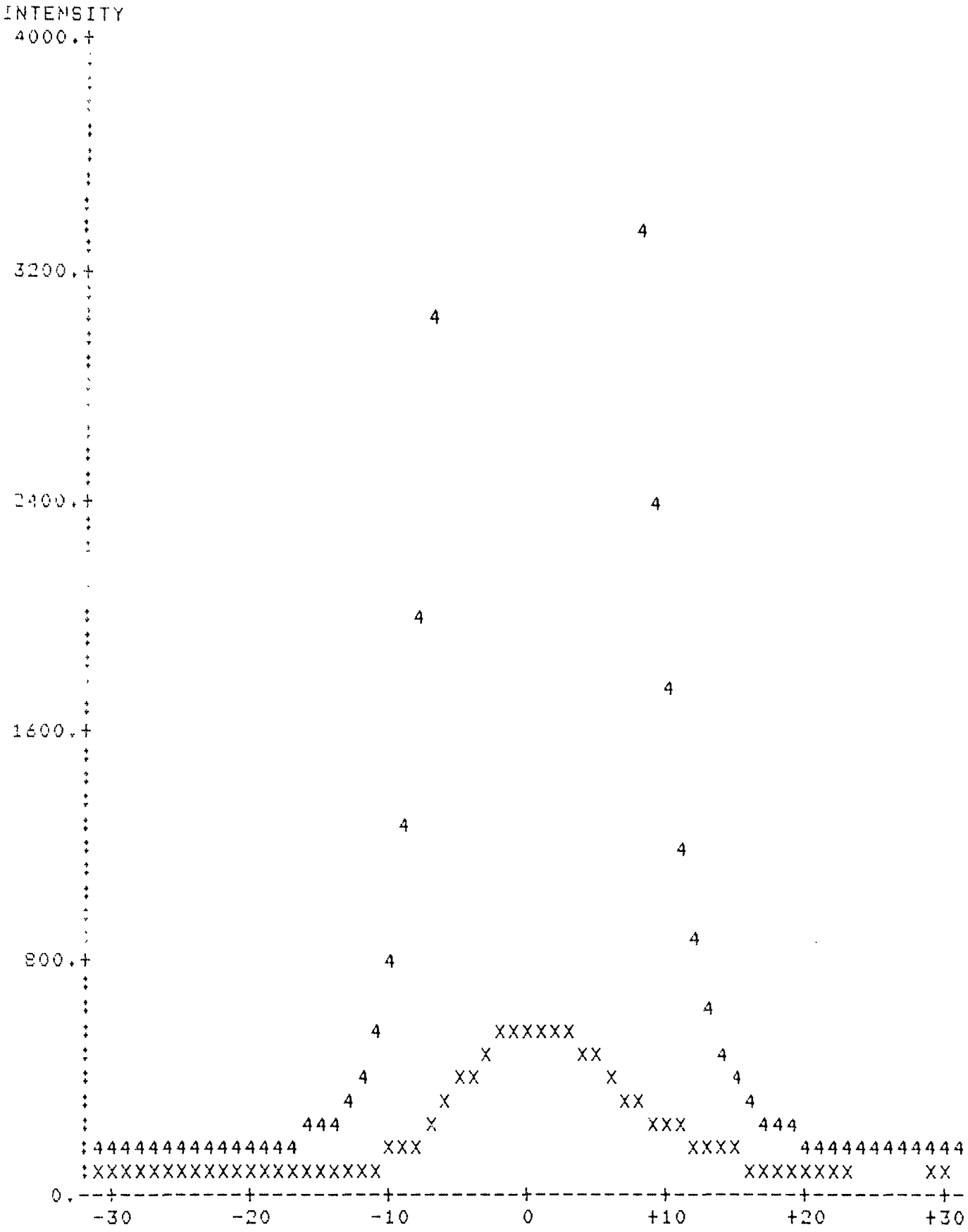

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WAVELENGTH SCANS FOR BA AT 4934 ANGSTROMS

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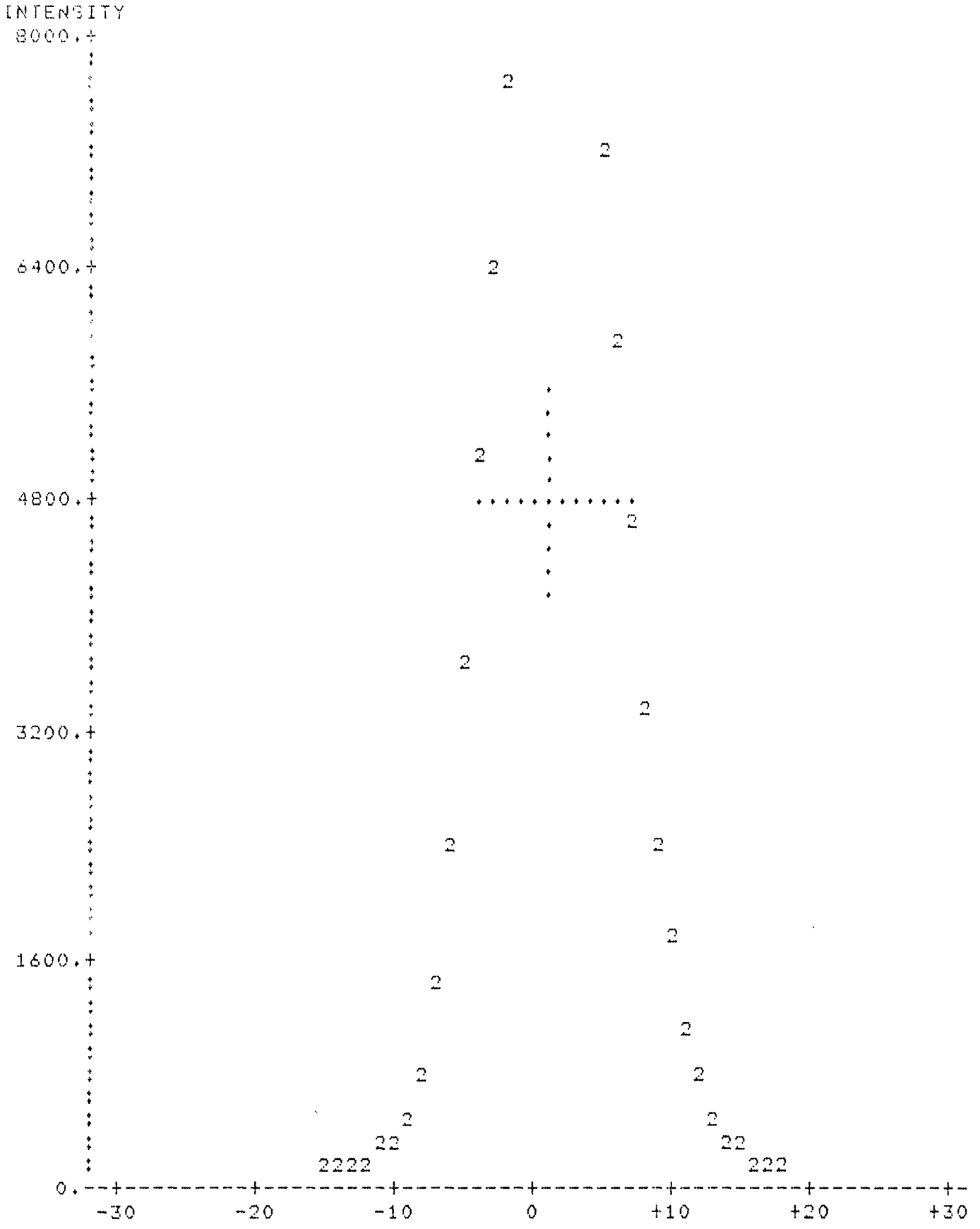

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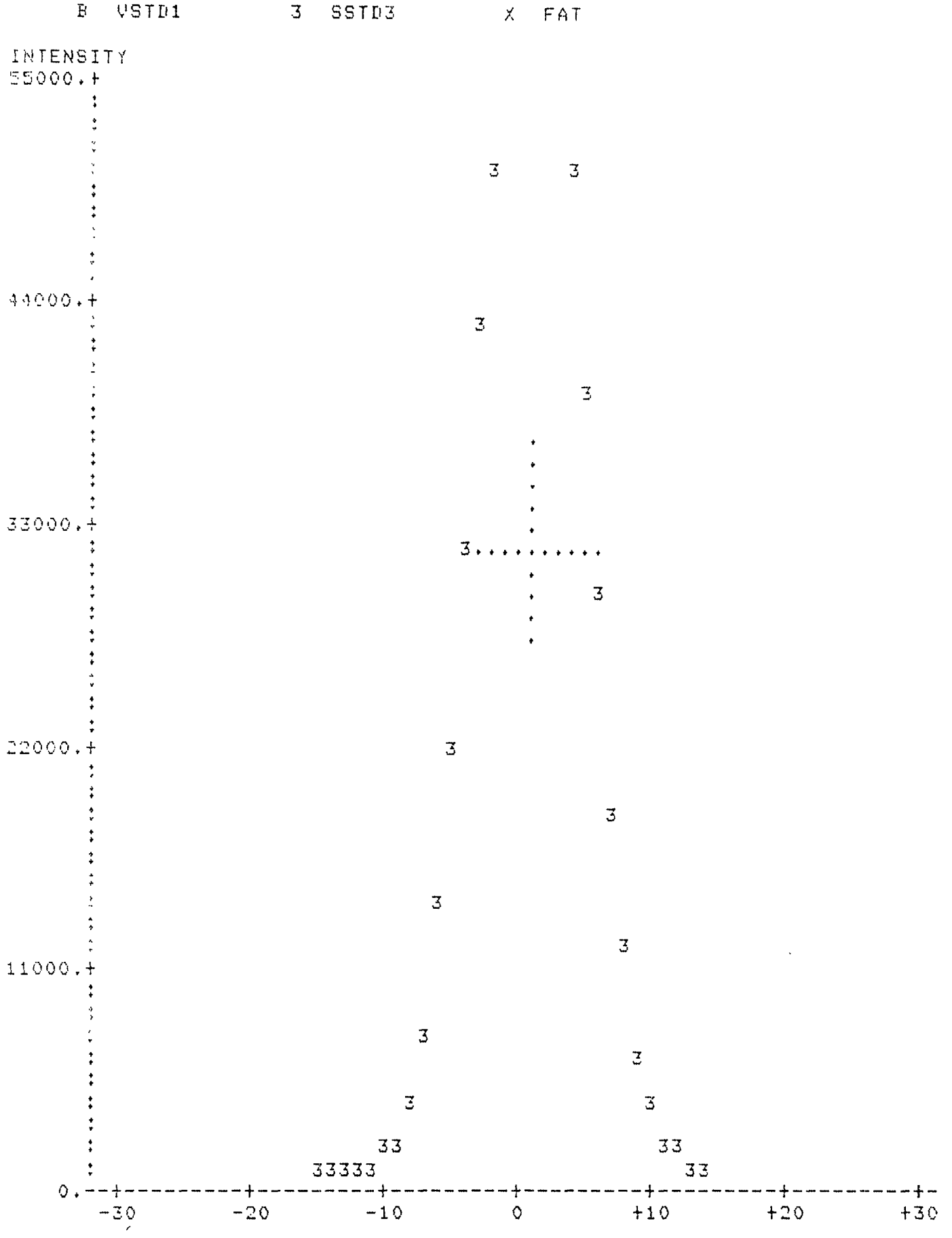
WAVELENGTH SCANS FOR BE AT 2348 ANGSTROMS



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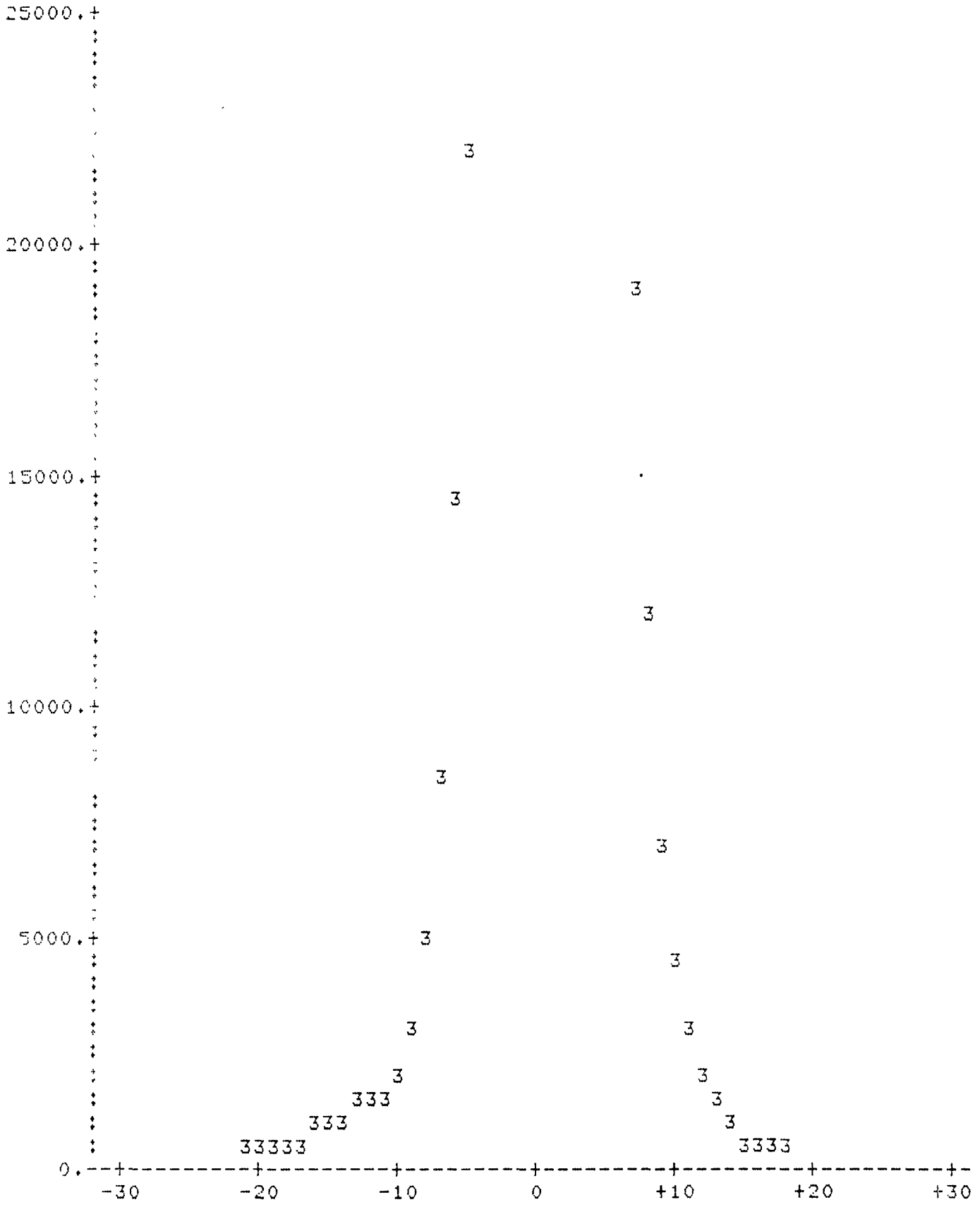

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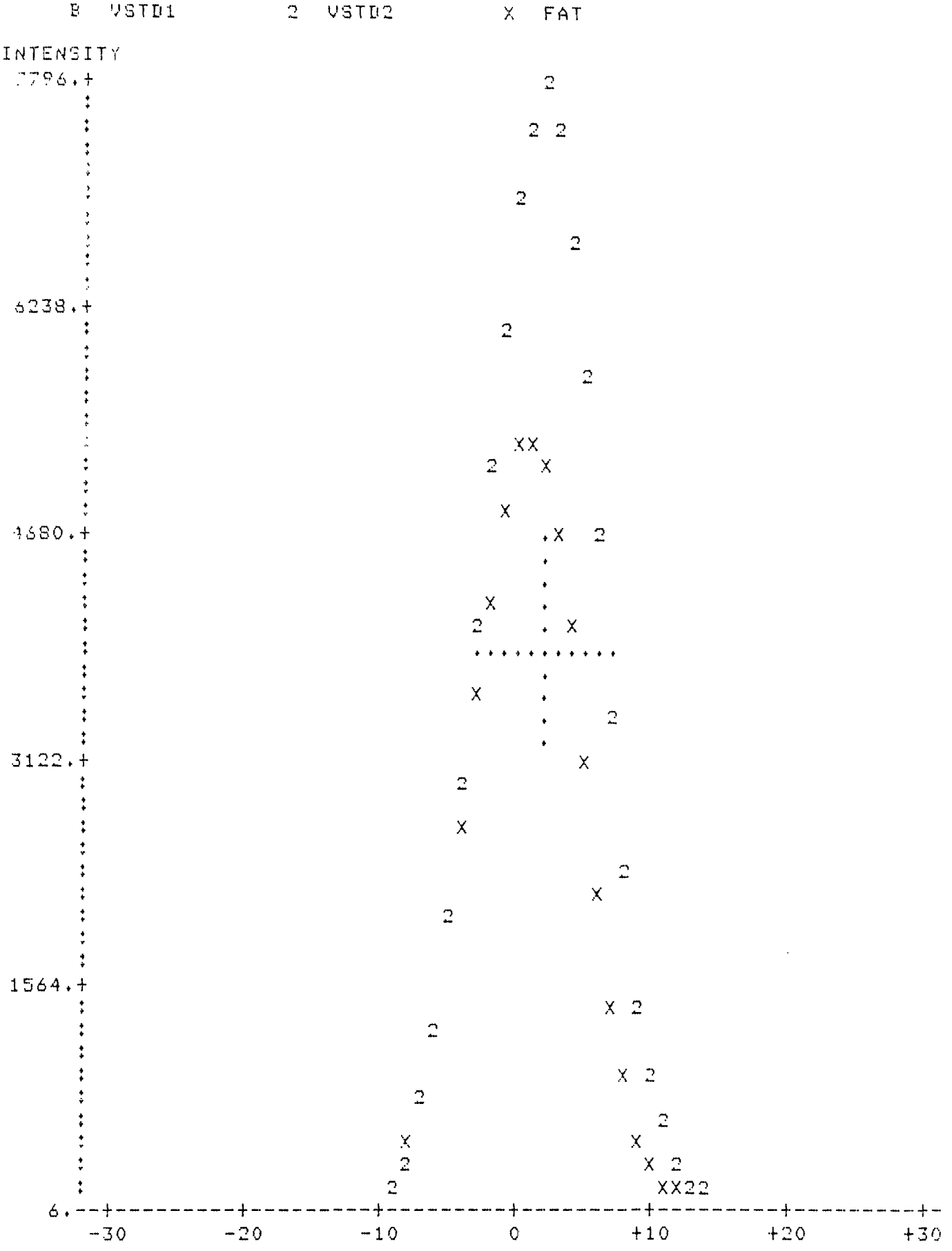
WAVELENGTH SCANS FOR CA AT 3968 ANGSTROMS



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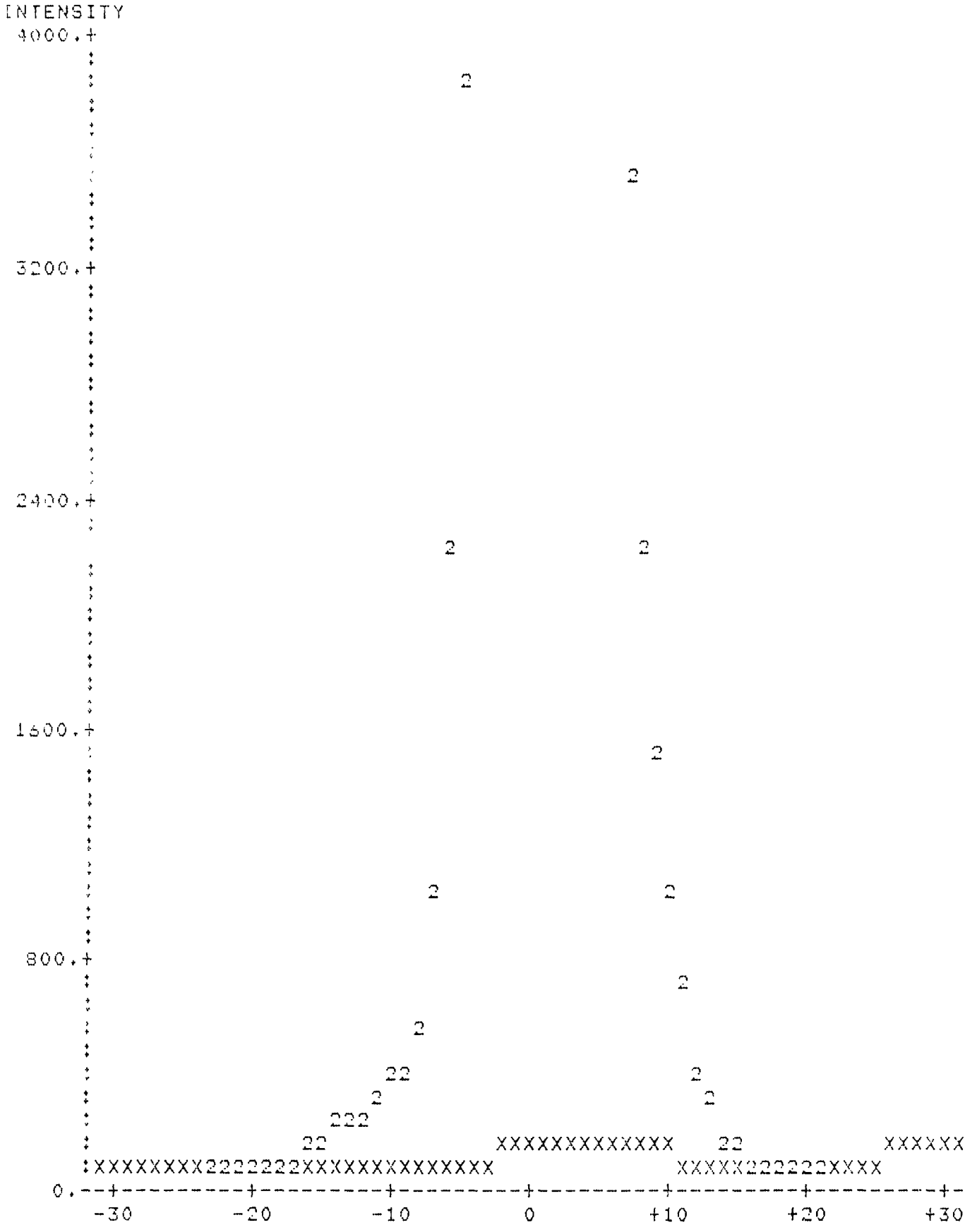

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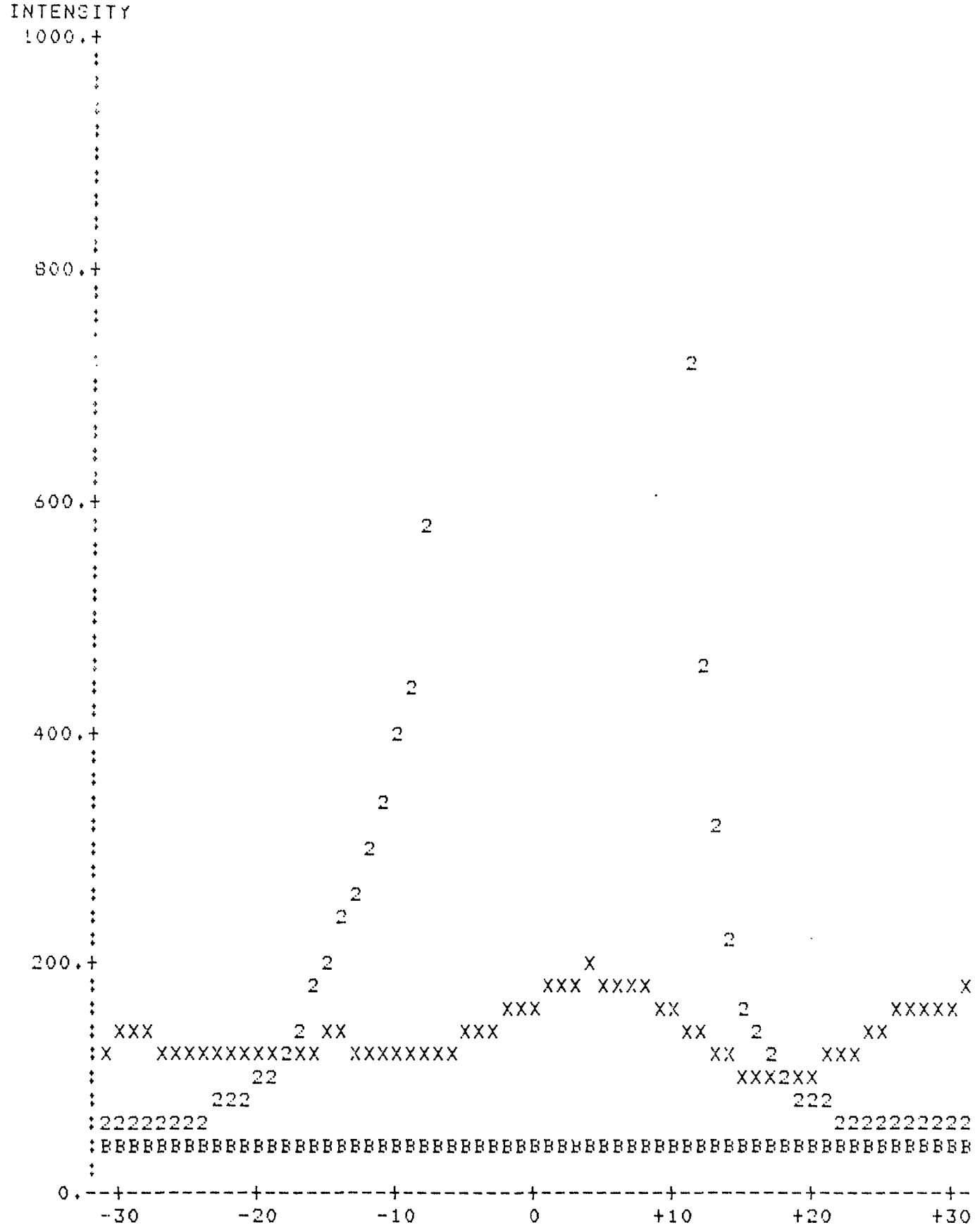

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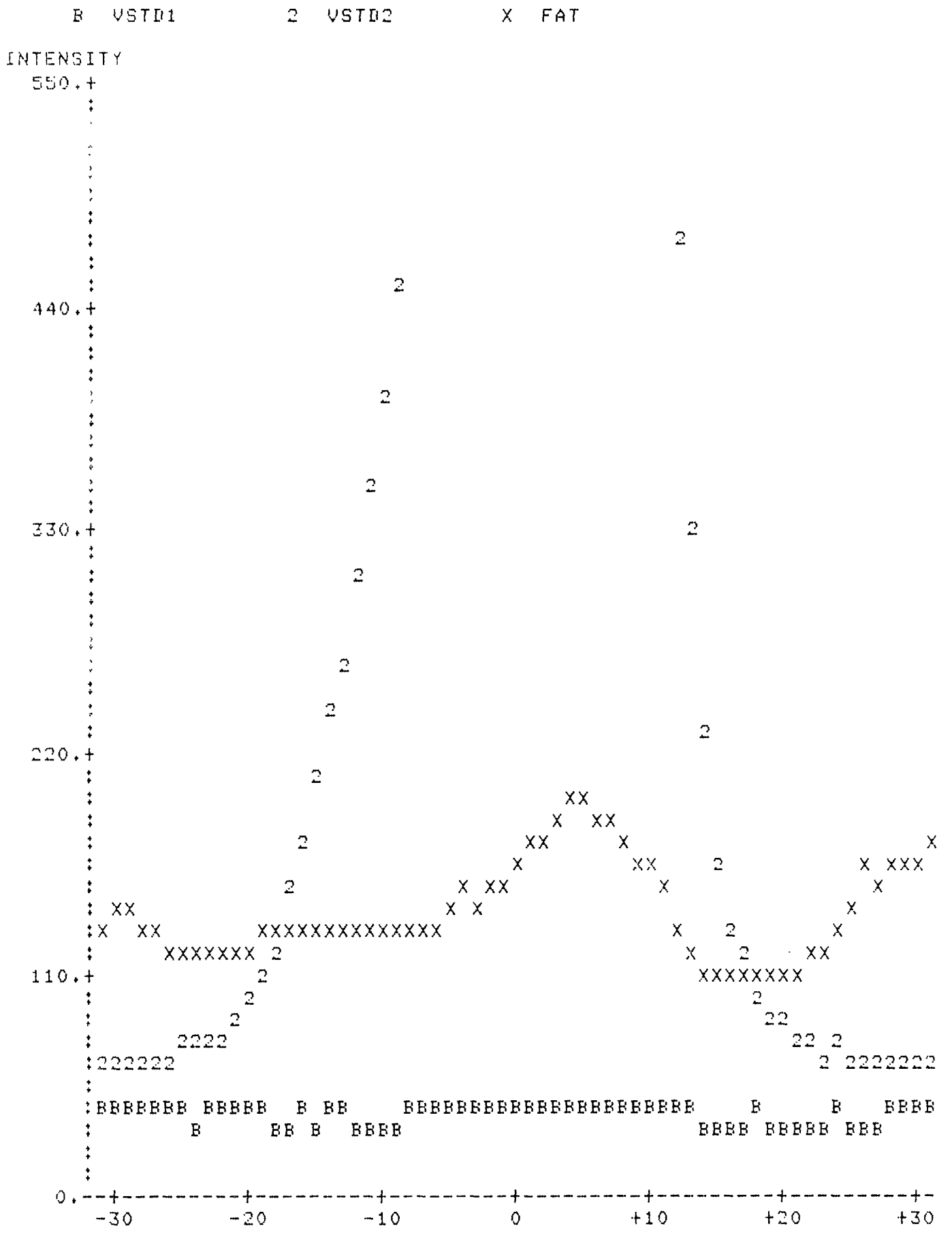
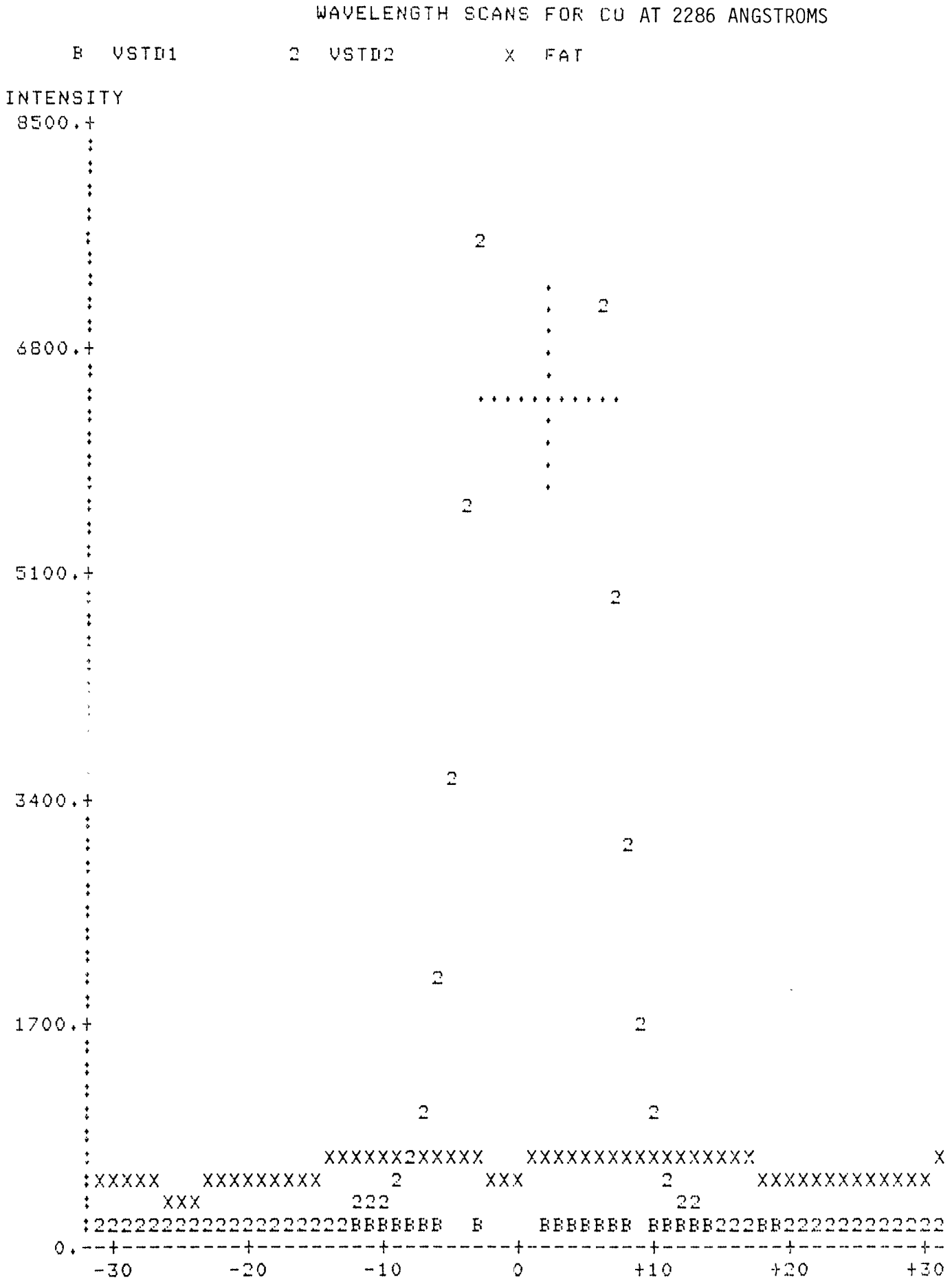
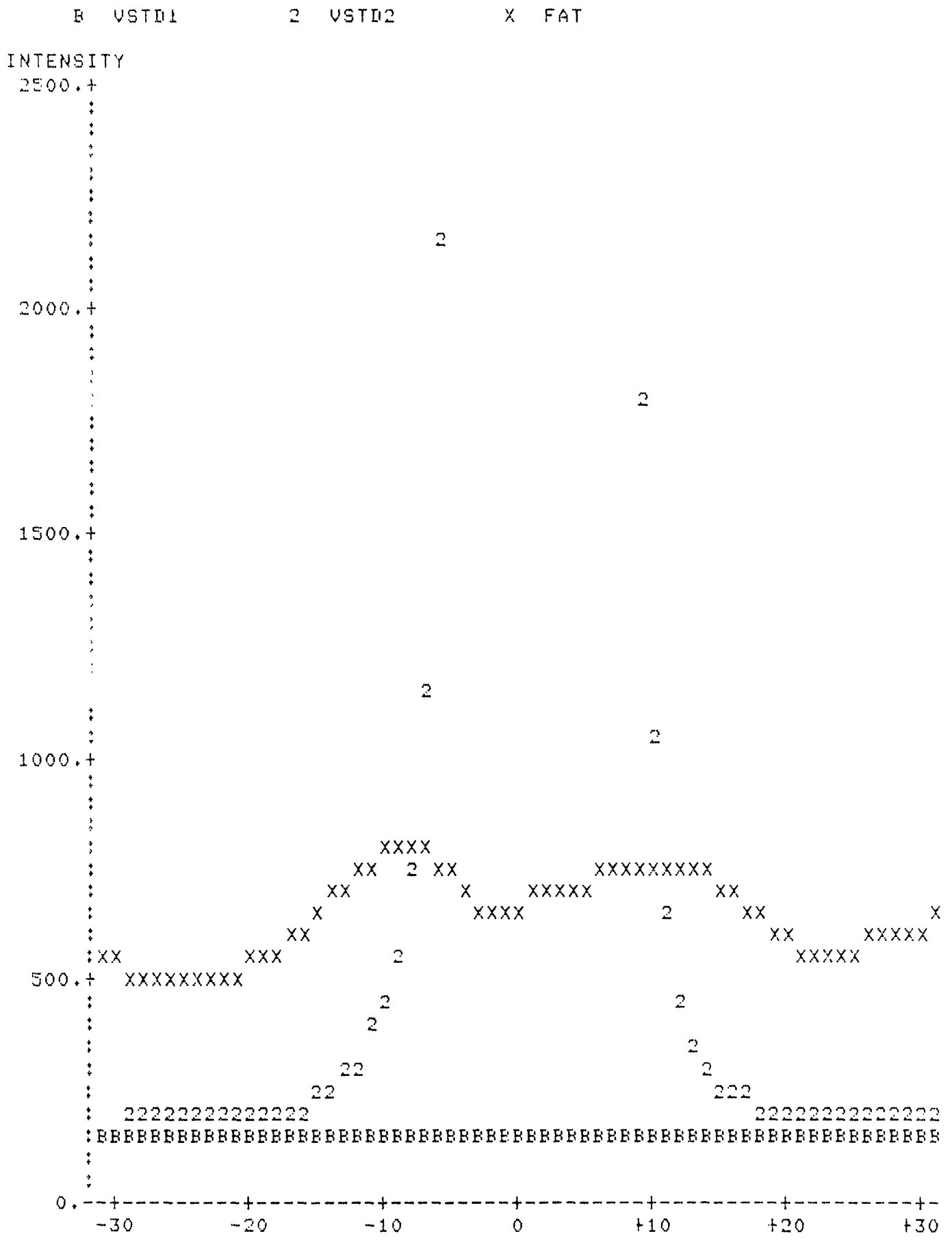


PEAK IS AT POSITION 1 HALF-WIDTH IS 10



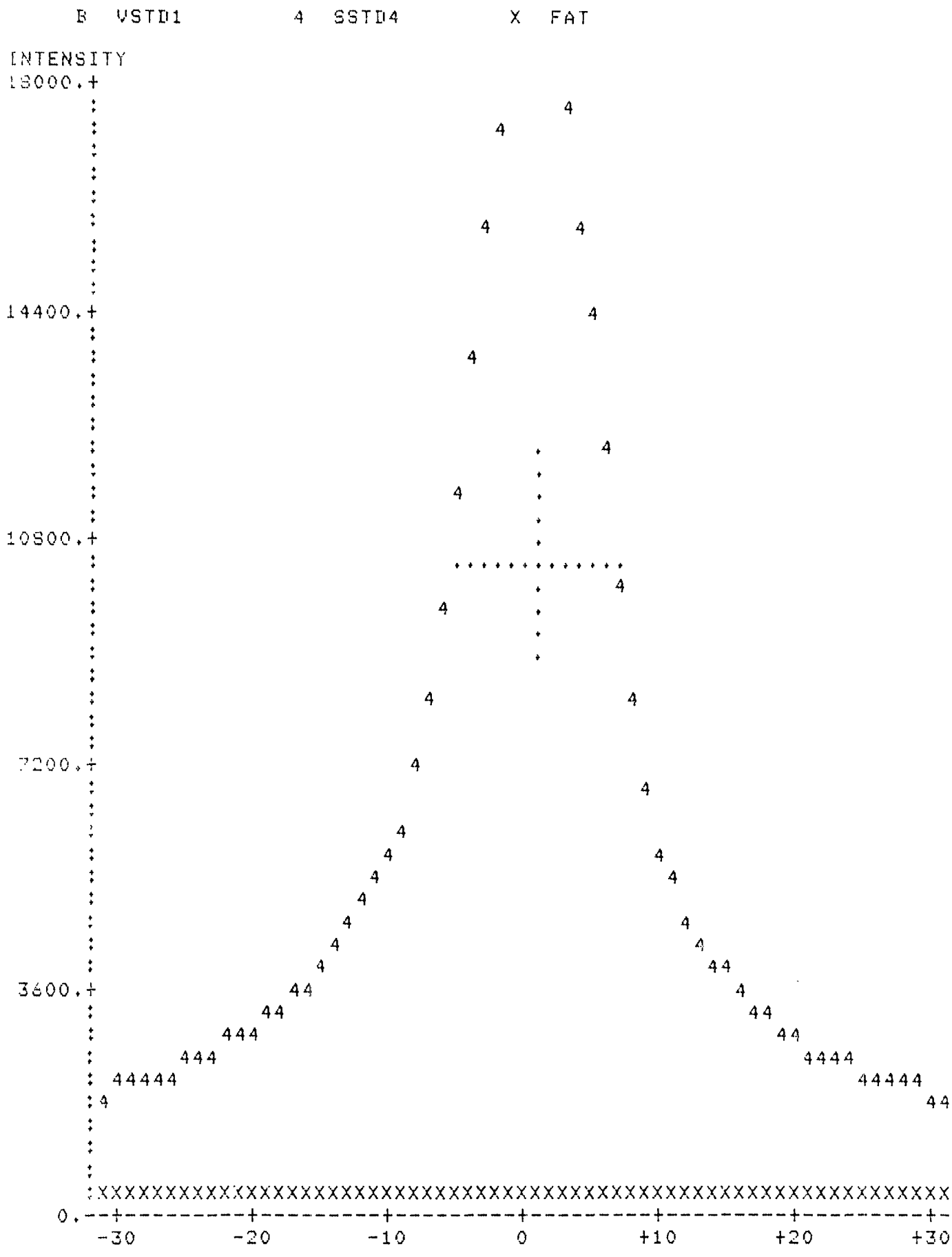
PEAK IS AT POSITION 2 HALF-WIDTH IS 11

WAVELENGTH SCANS FOR CO AT 2286 ANGSTROMS



PEAK IS AT POSITION 2 HALF-WIDTH IS 11

WAVELENGTH SCANS FOR CR AT 2677 ANGSTROMS



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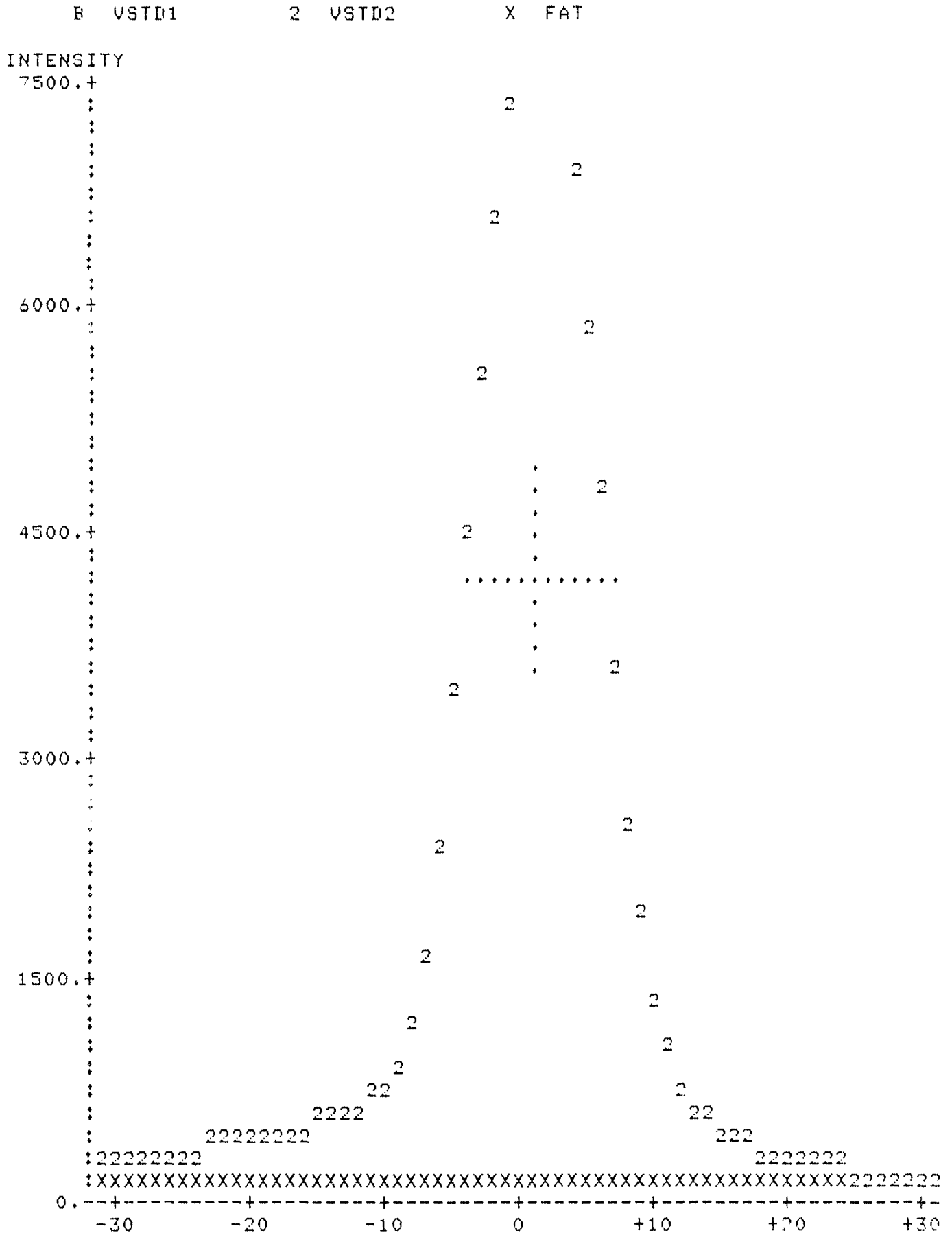
WAVELENGTH SCANS FOR CU AT 3247 ANGSTROMS



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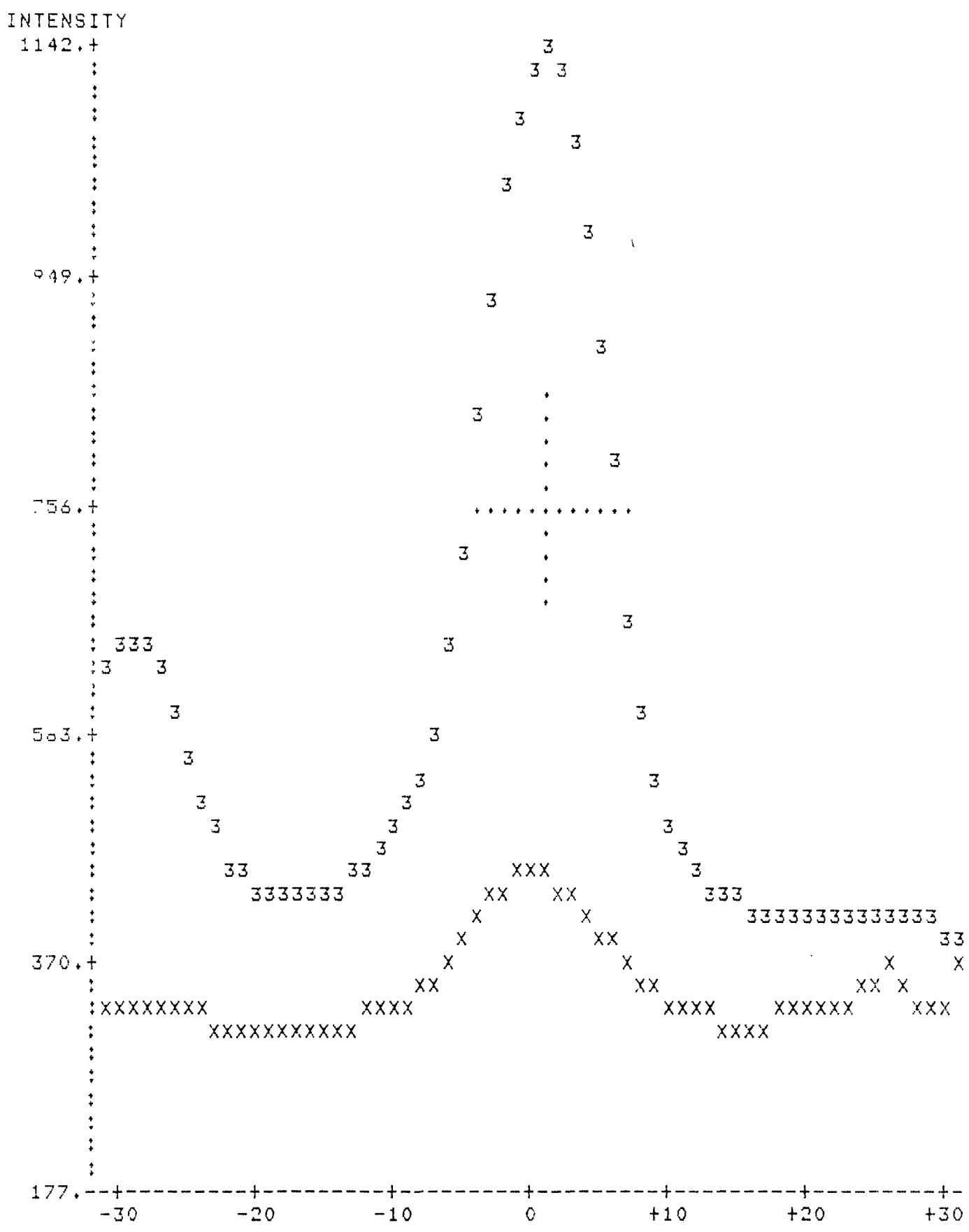
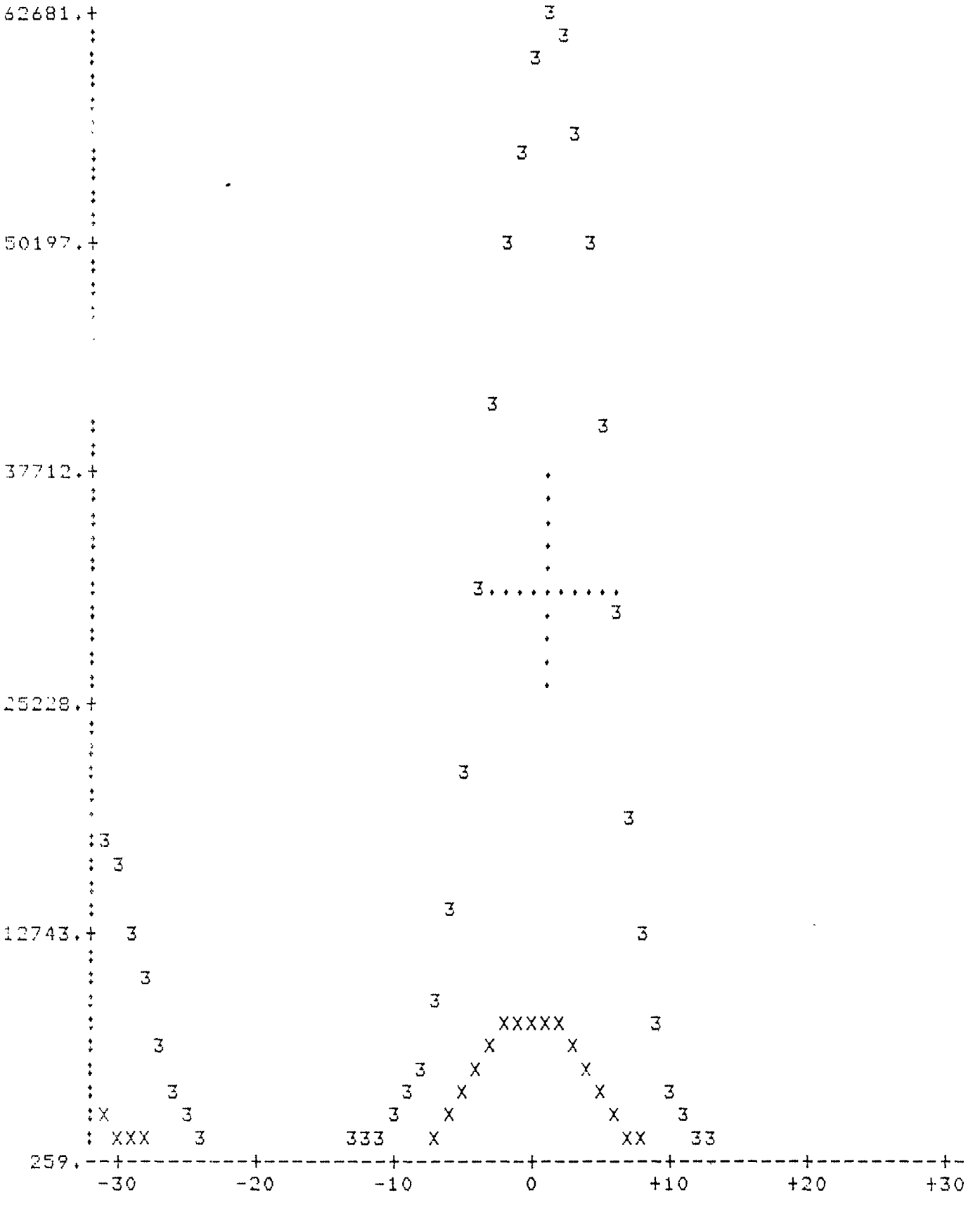
WAVELENGTH SCANS FOR FE AT 2714 ANGSTROMS



PEAK IS AT POSITION 1 HALF-WIDTH IS 12

WAVELENGTH SCANS FOR FE AT 2599 ANGSTROMS

B VSTD1 3 SSTD3 X FAT



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WAVELENGTH SCANS FOR HG AT 1942 ANGSTROMS

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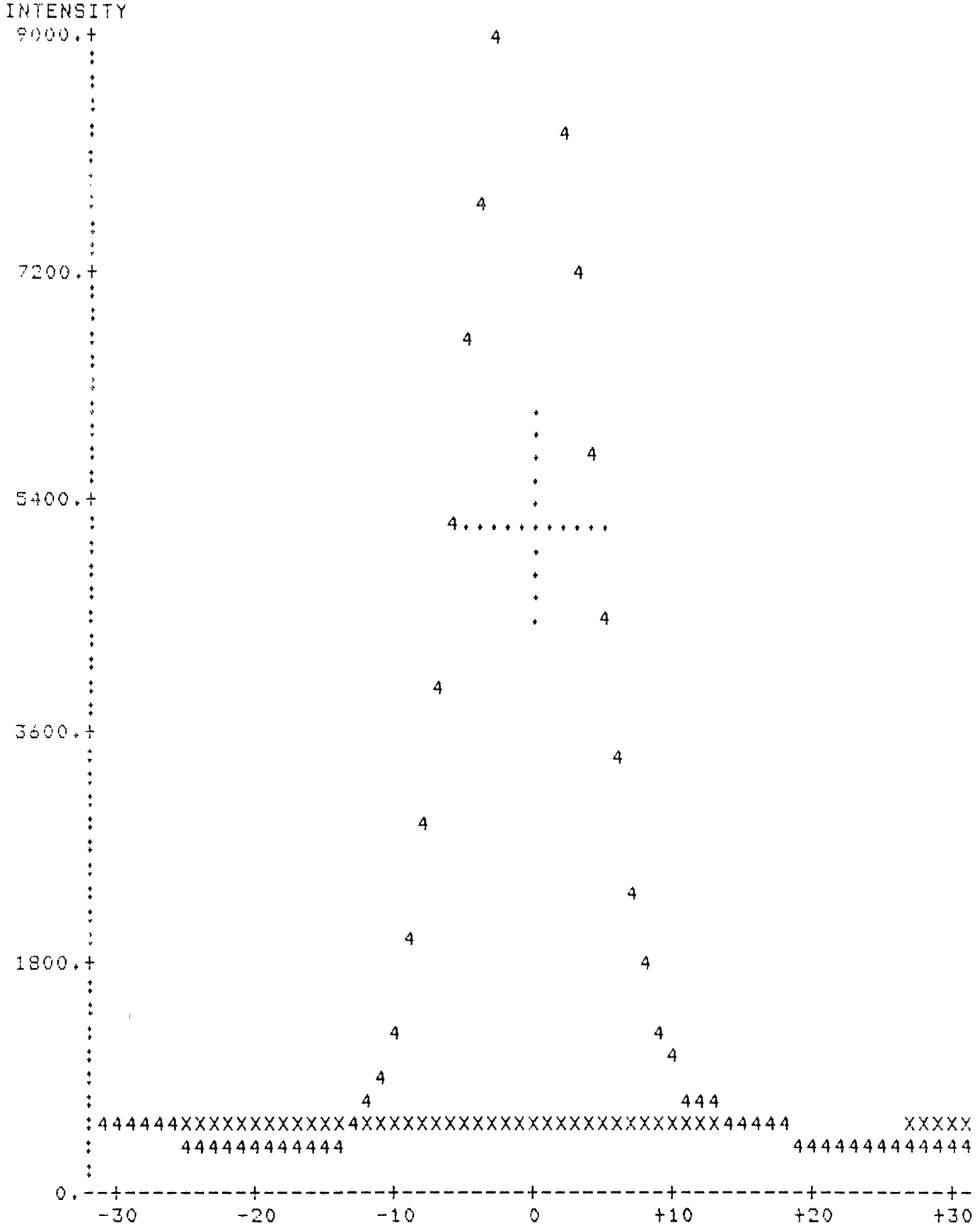

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WAVELENGTH SCANS FOR HG AT 1942 ANGSTROMS

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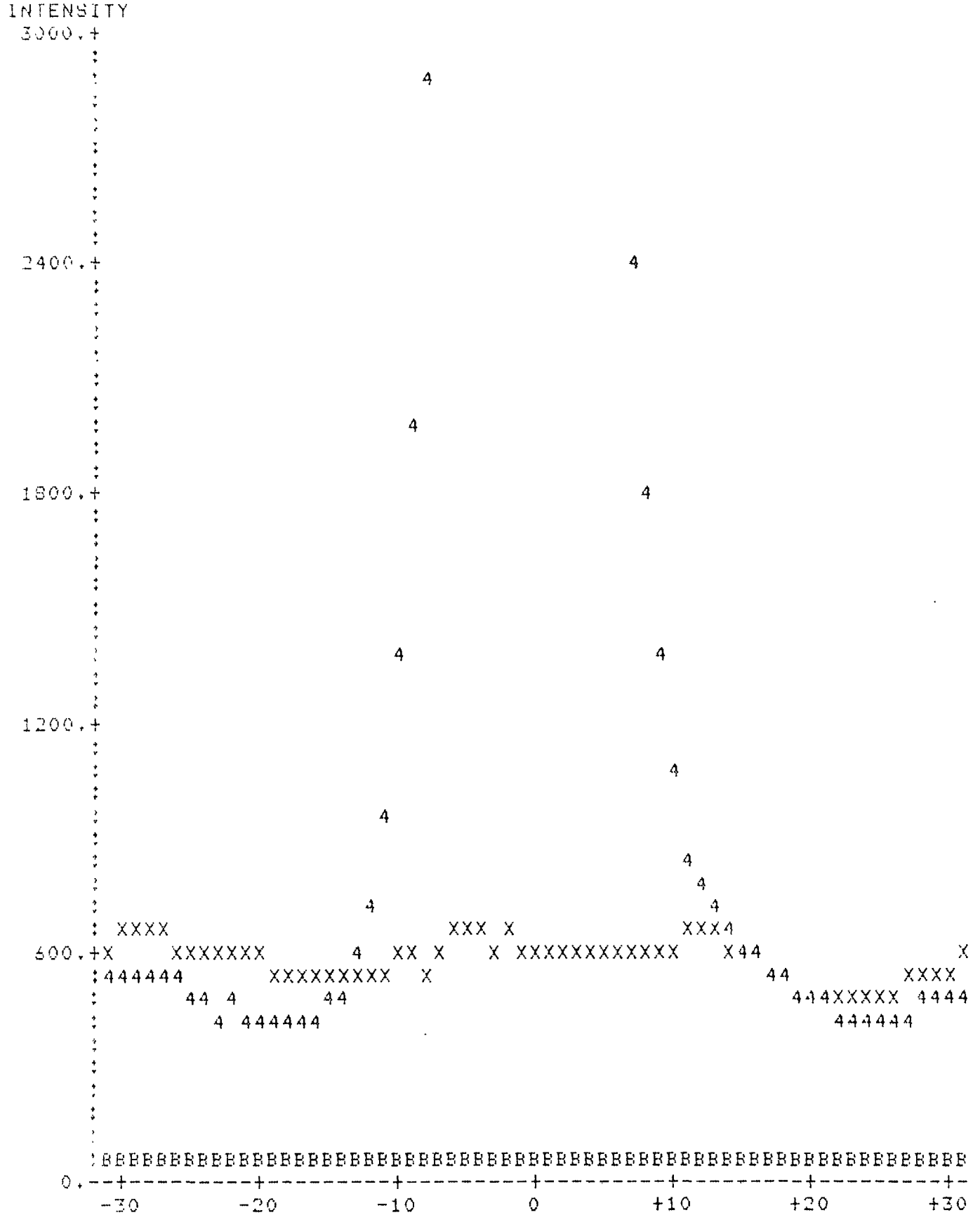
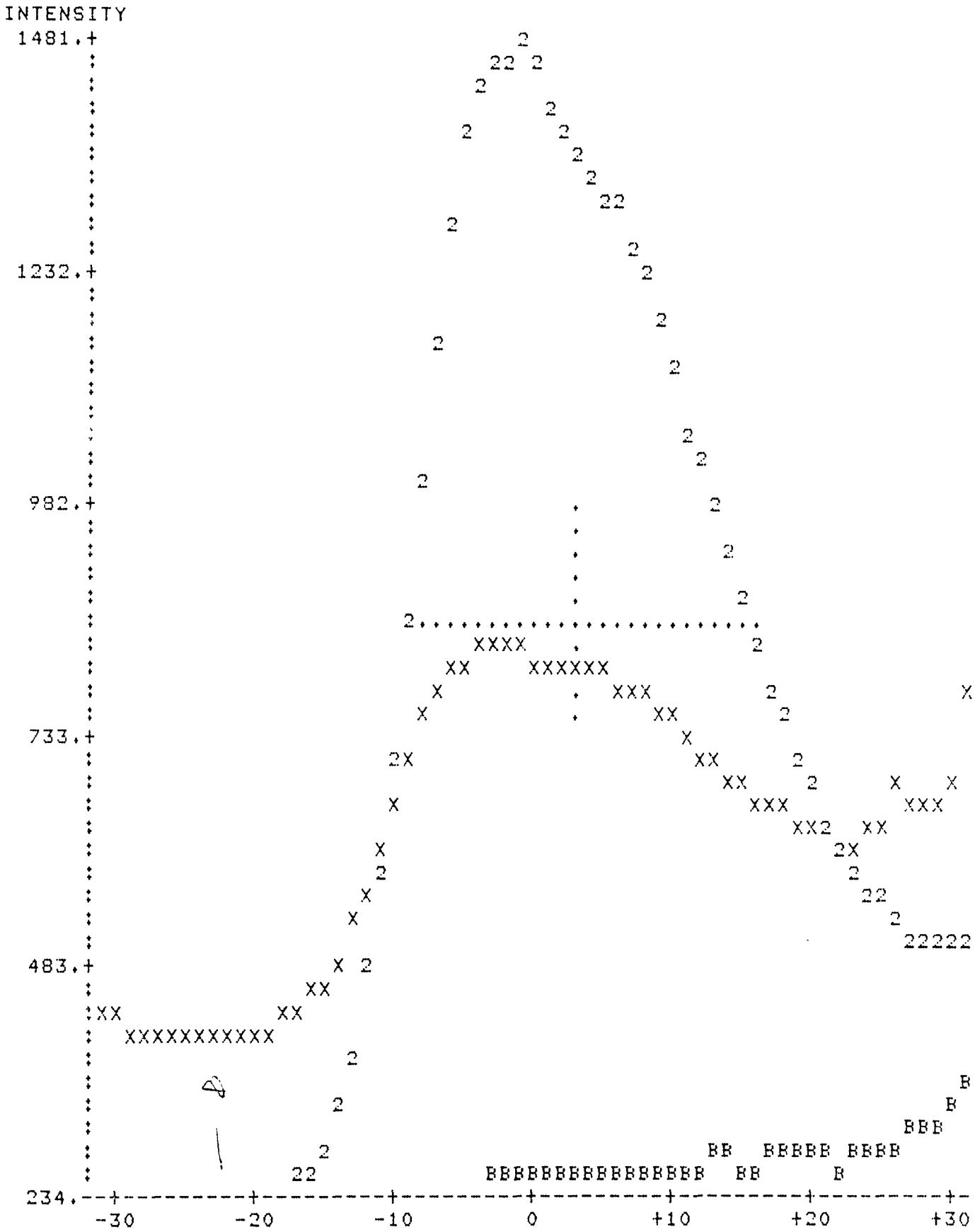


PEAK IS AT POSITION 0 HALF-WIDTH IS 11

WAVELENGTH SCANS FOR K AT 7664 ANGSTROMS

B VSTD1 2 VSTD2 X FAT



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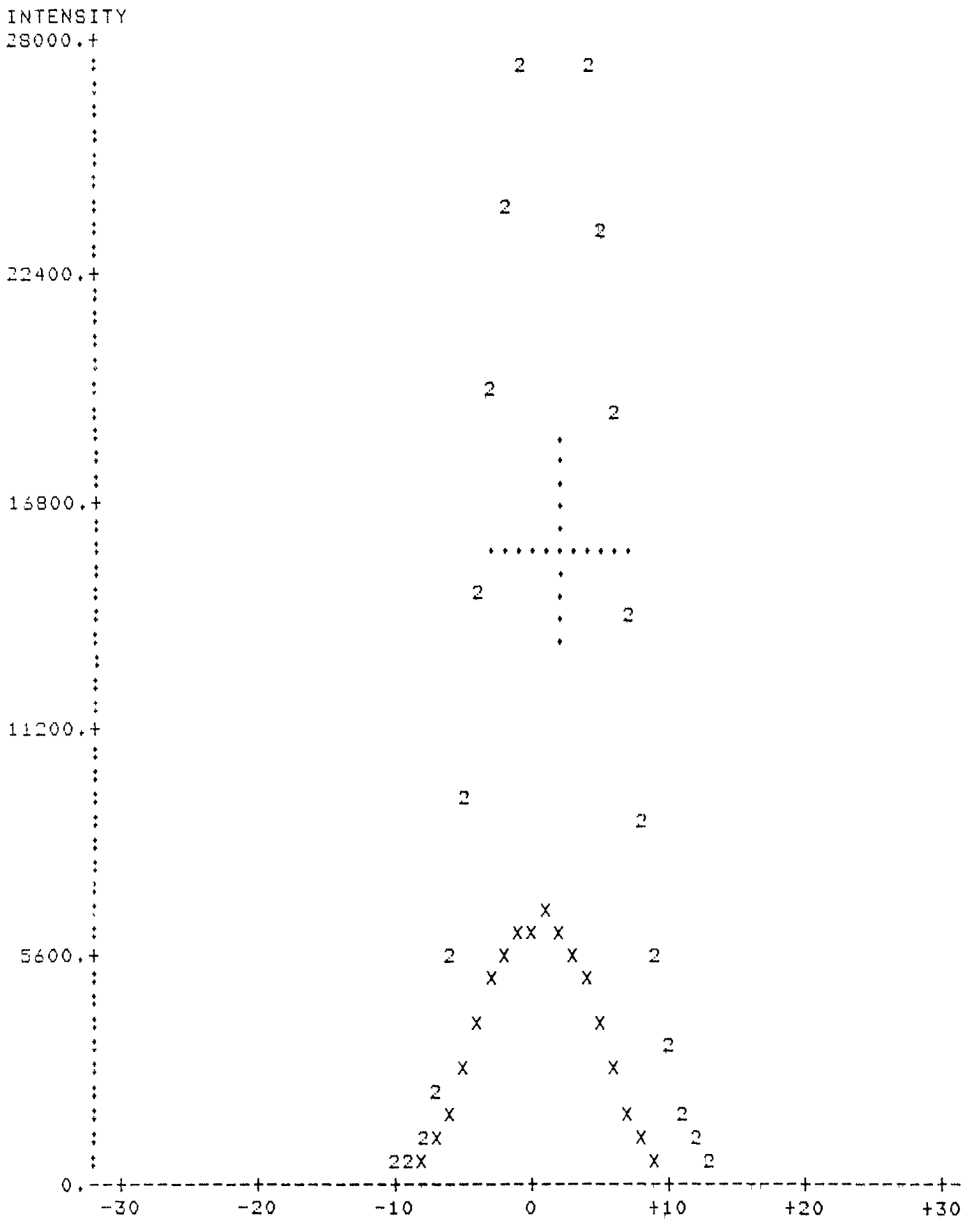
WAVELENGTH SCANS FOR MG AT 2795 ANGSTROMS



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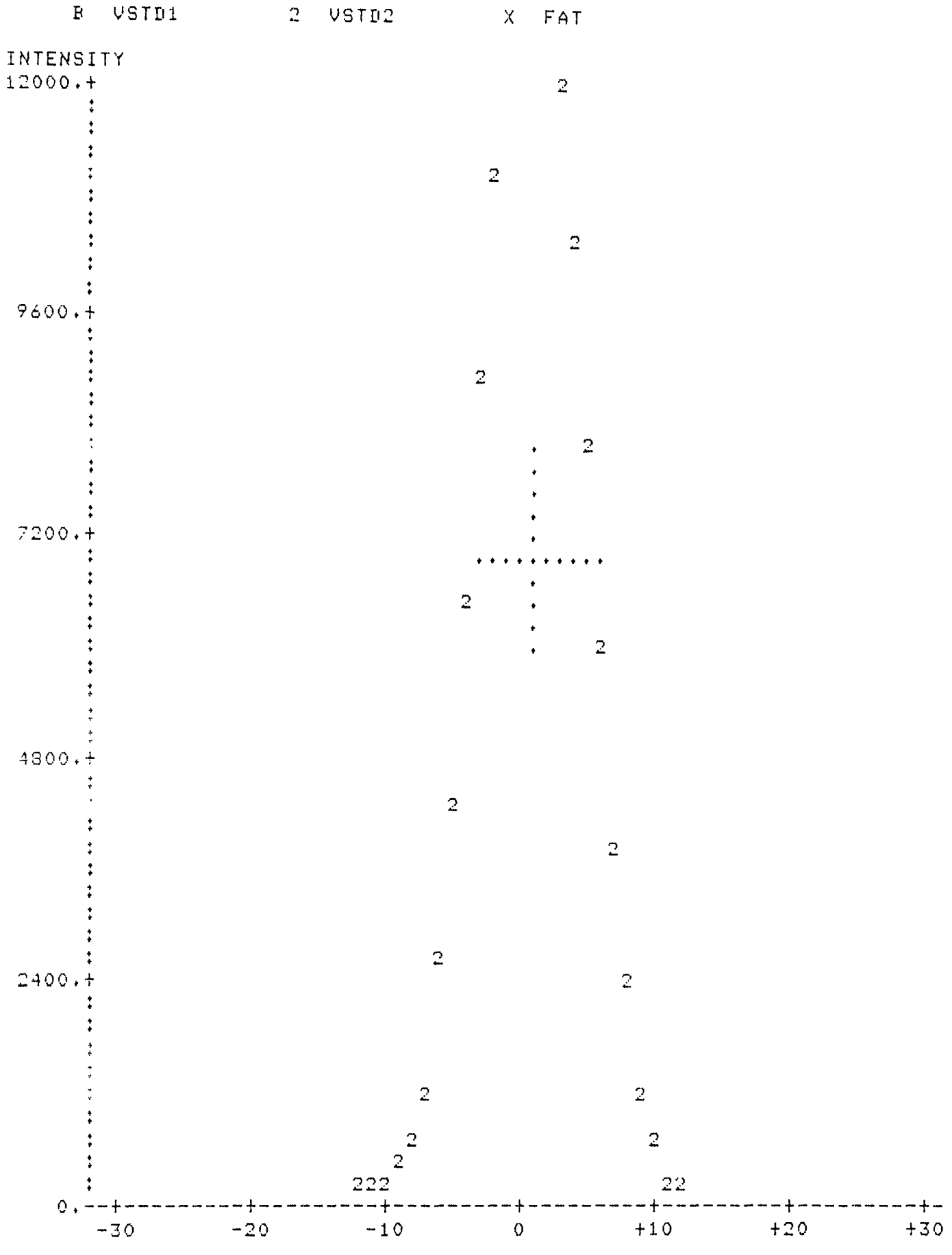
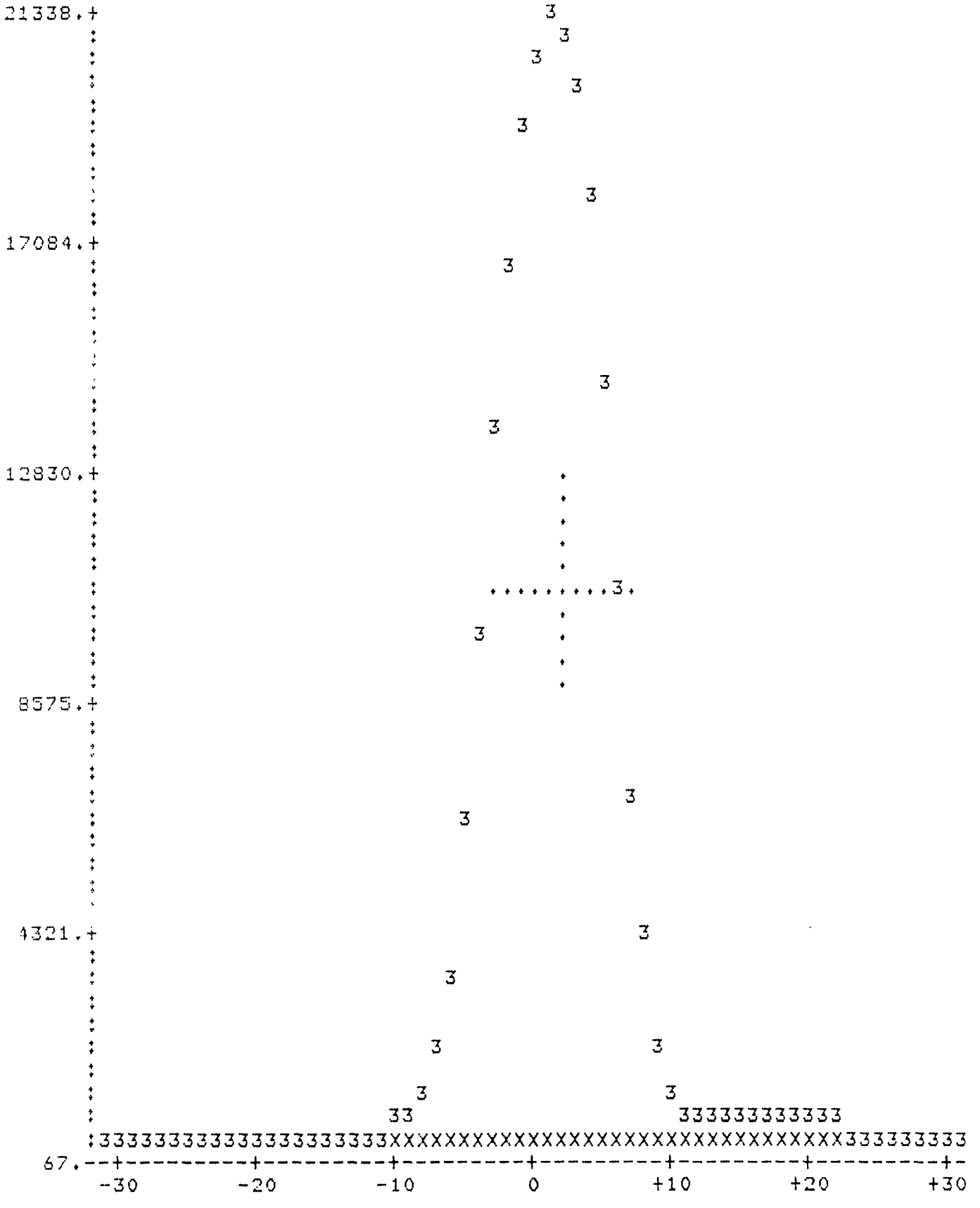
WAVELENGTH SCANS FOR MN AT 2576 ANGSTROMS



PEAK IS AT POSITION 1 HALF-WIDTH IS 10

WAVELENGTH SCANS FOR MO AT 2020 ANGSTROMS

B VSTD1 3 SSTD3 X FAT



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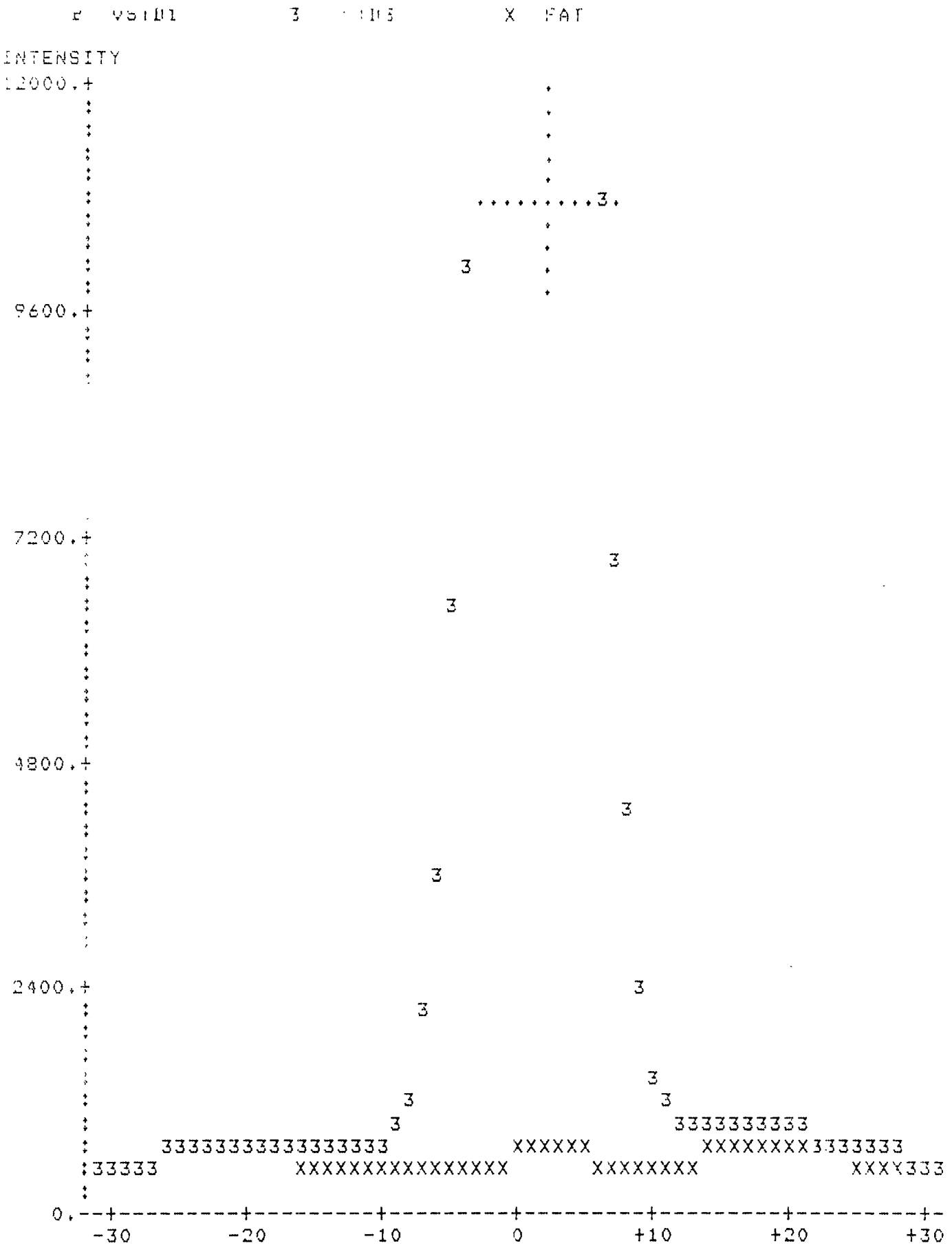
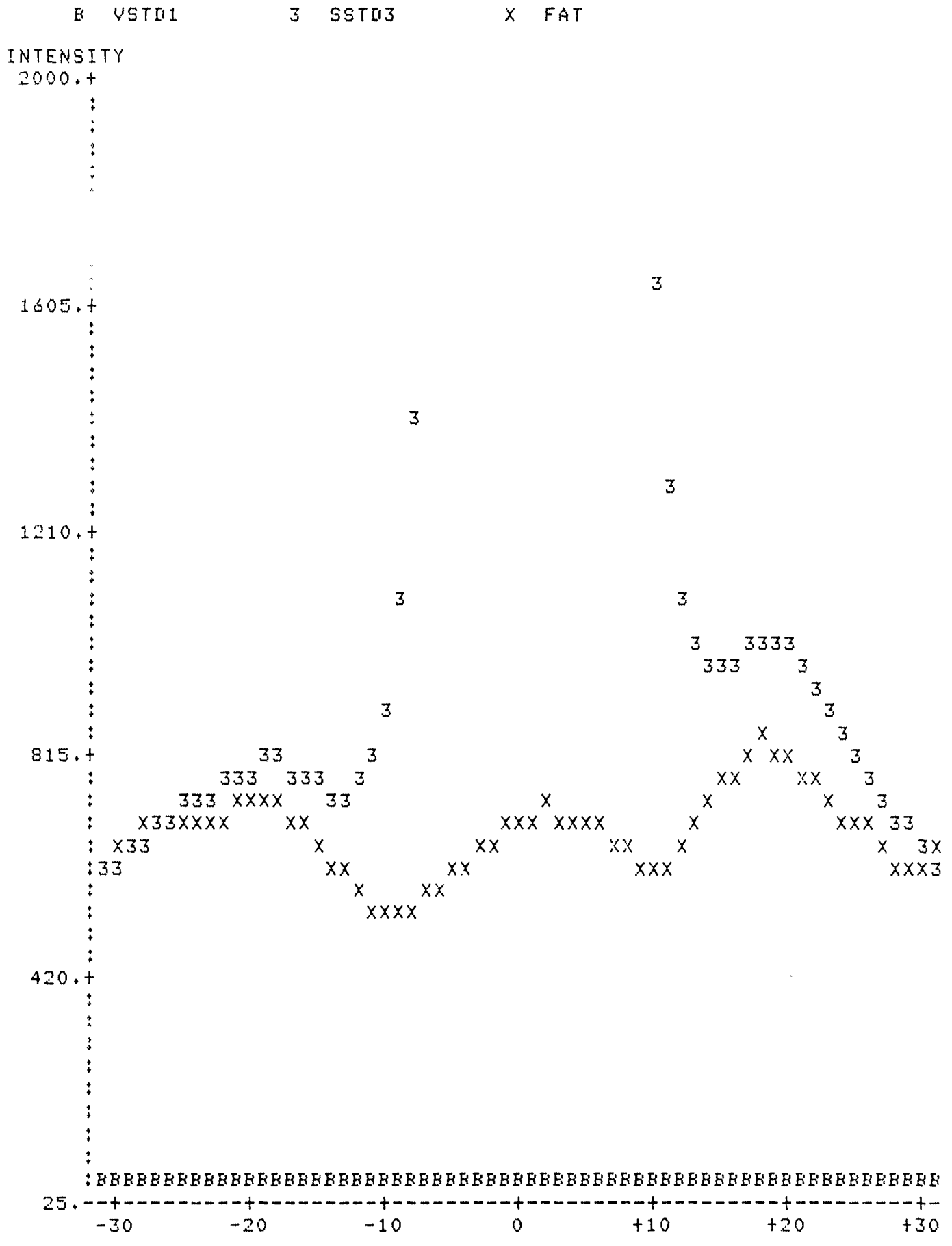


PEAK IS AT POSITION 2 HALF-WIDTH IS 11

WAVELENGTH SCANS FOR MO AT 2020 ANGSTROMS



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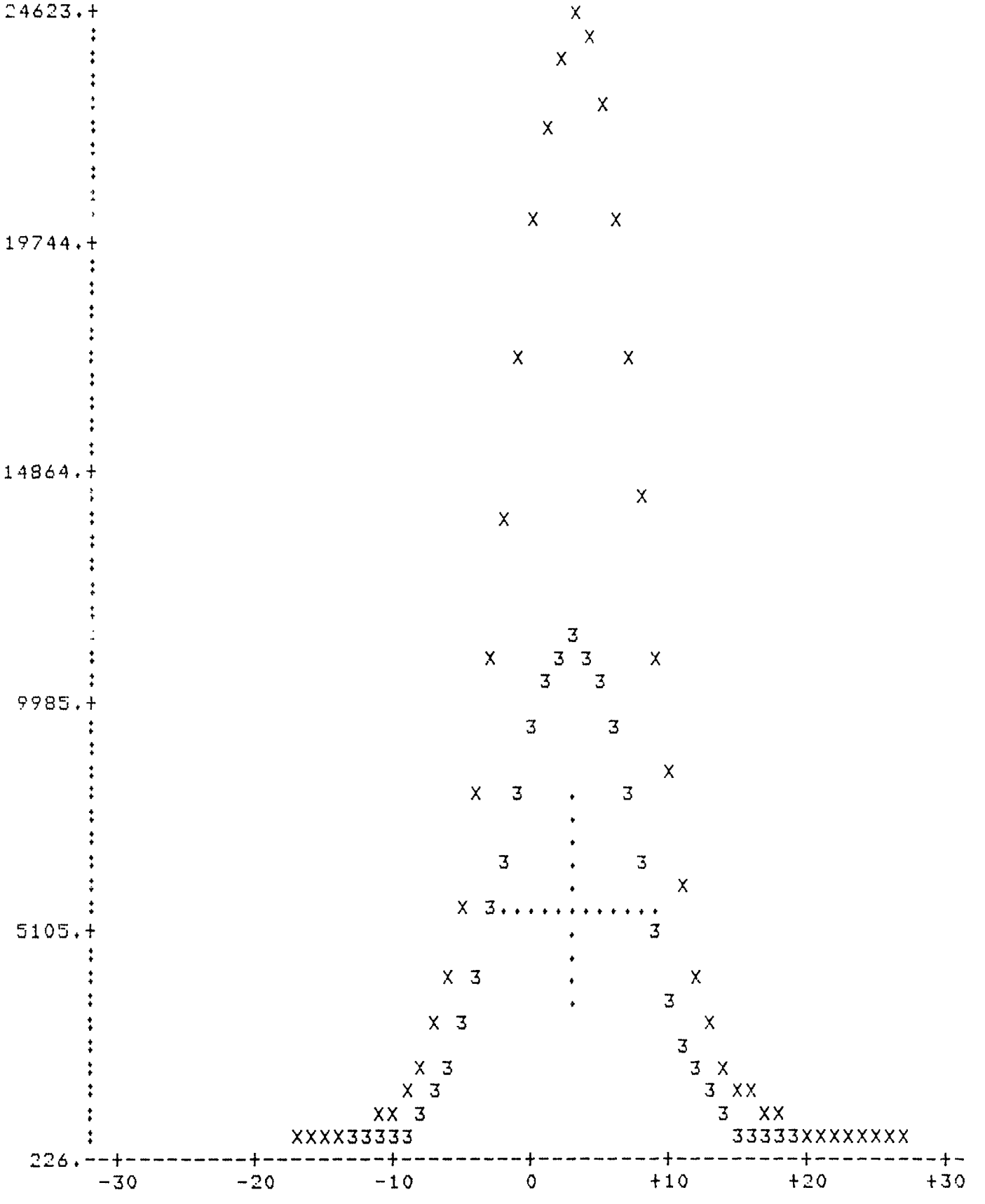
WAVELENGTH SCANS FOR NA AT 5890 ANGSTROMS

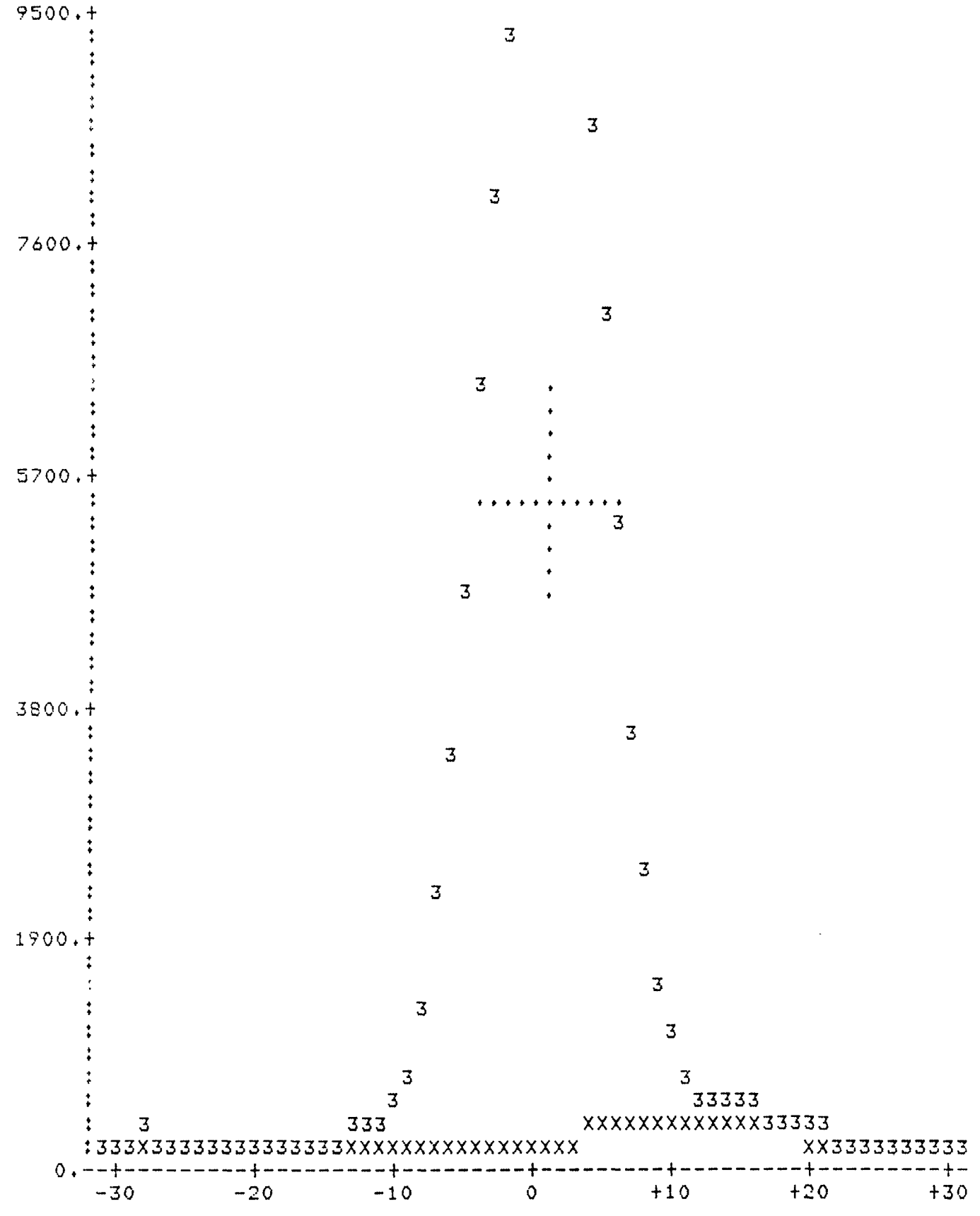
B VSTD1 3 SSTD3 X FAT



PEAK IS AT POSITION 3 HALF-WIDTH IS 12

WAVELENGTH SCANS FOR NI AT 2316 ANGSTROMS

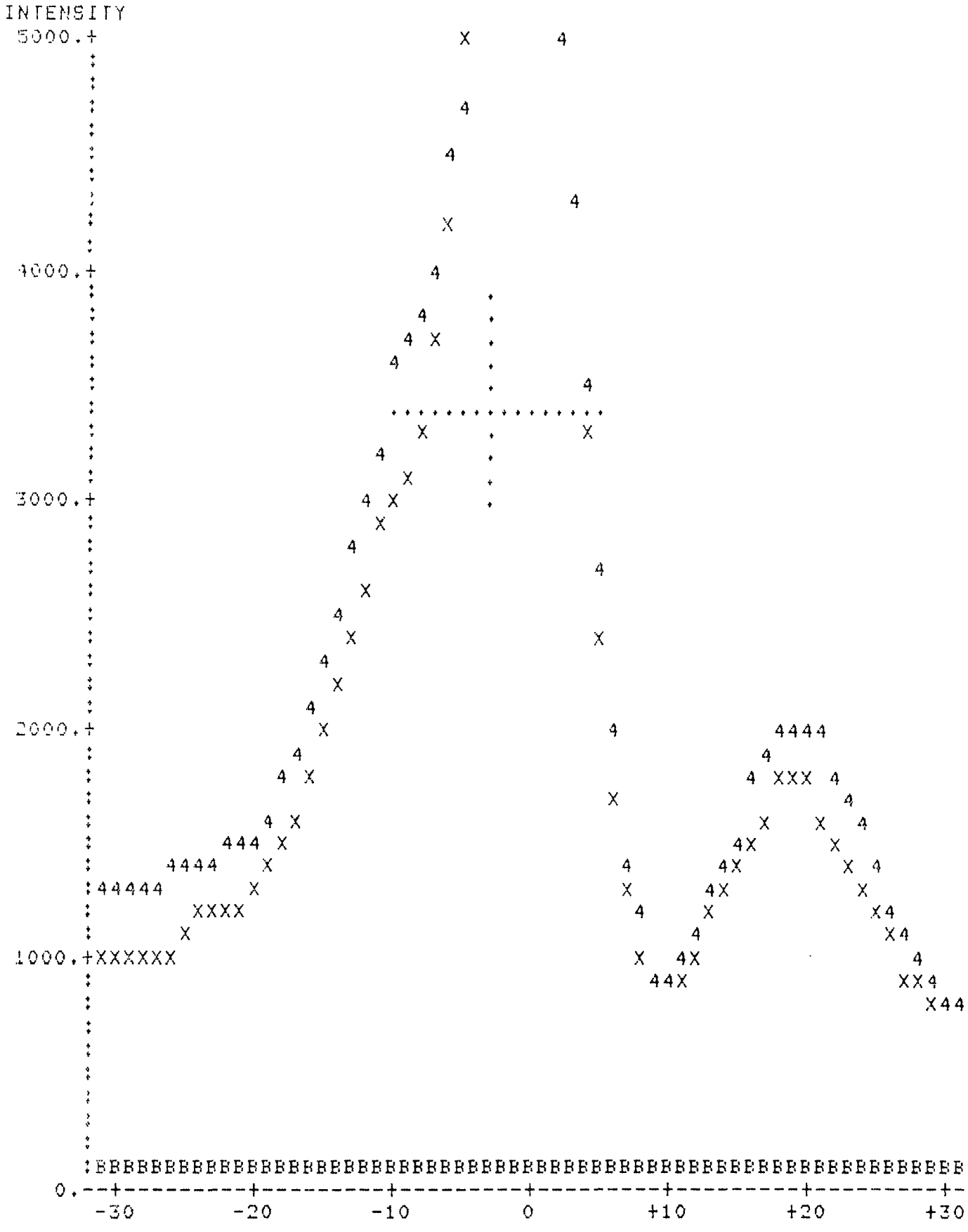
B VSTD1 3 SSTD3 X FAT



PEAK IS AT POSITION 0 HALF-WIDTH IS 10

WAVELENGTH SCANS FOR F AT 2149 ANGSTROMS

B VSTD1 4 SSTD4 X FAT



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WAVELENGTH SCANS FOR P AT 2149 ANGSTROMS

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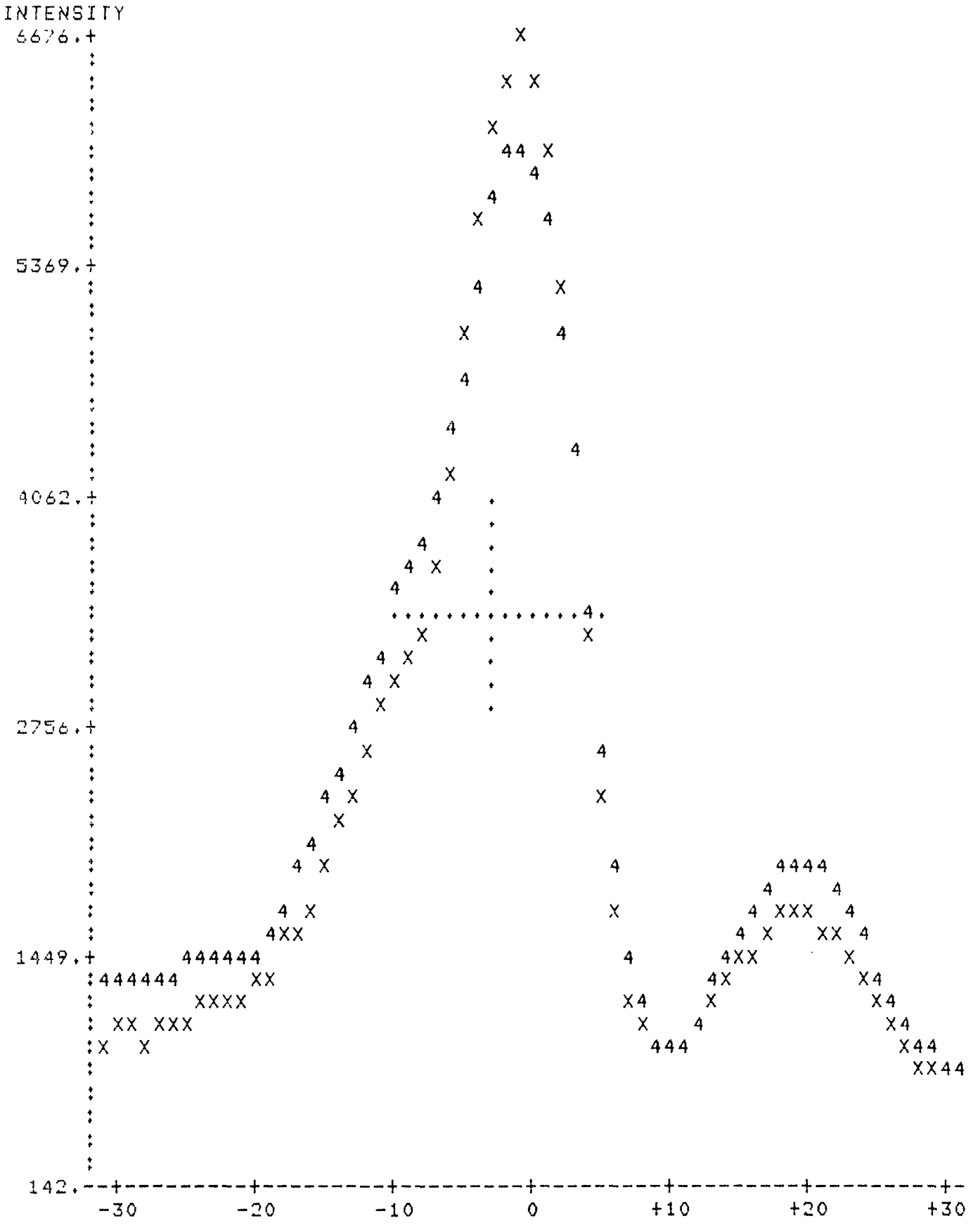

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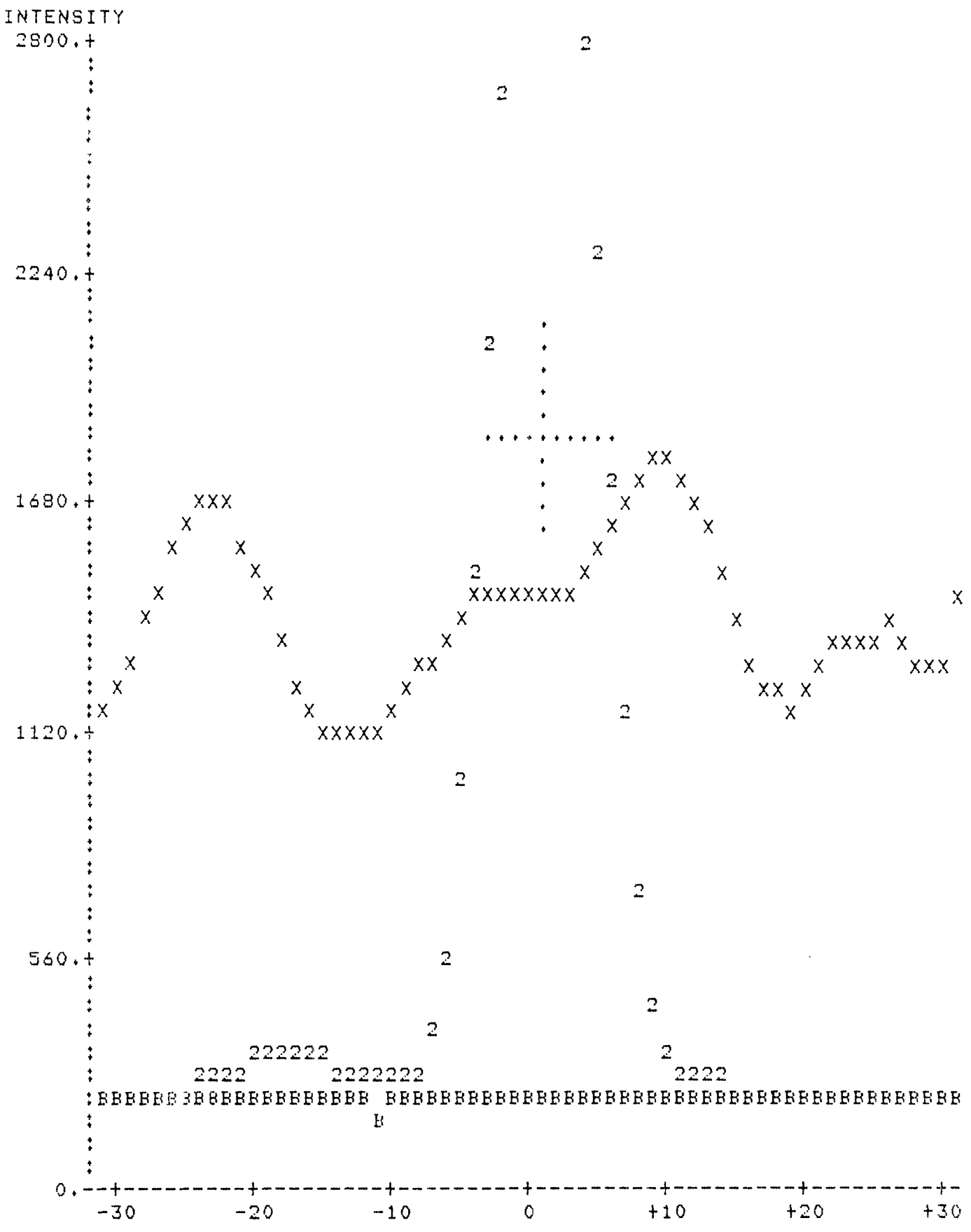
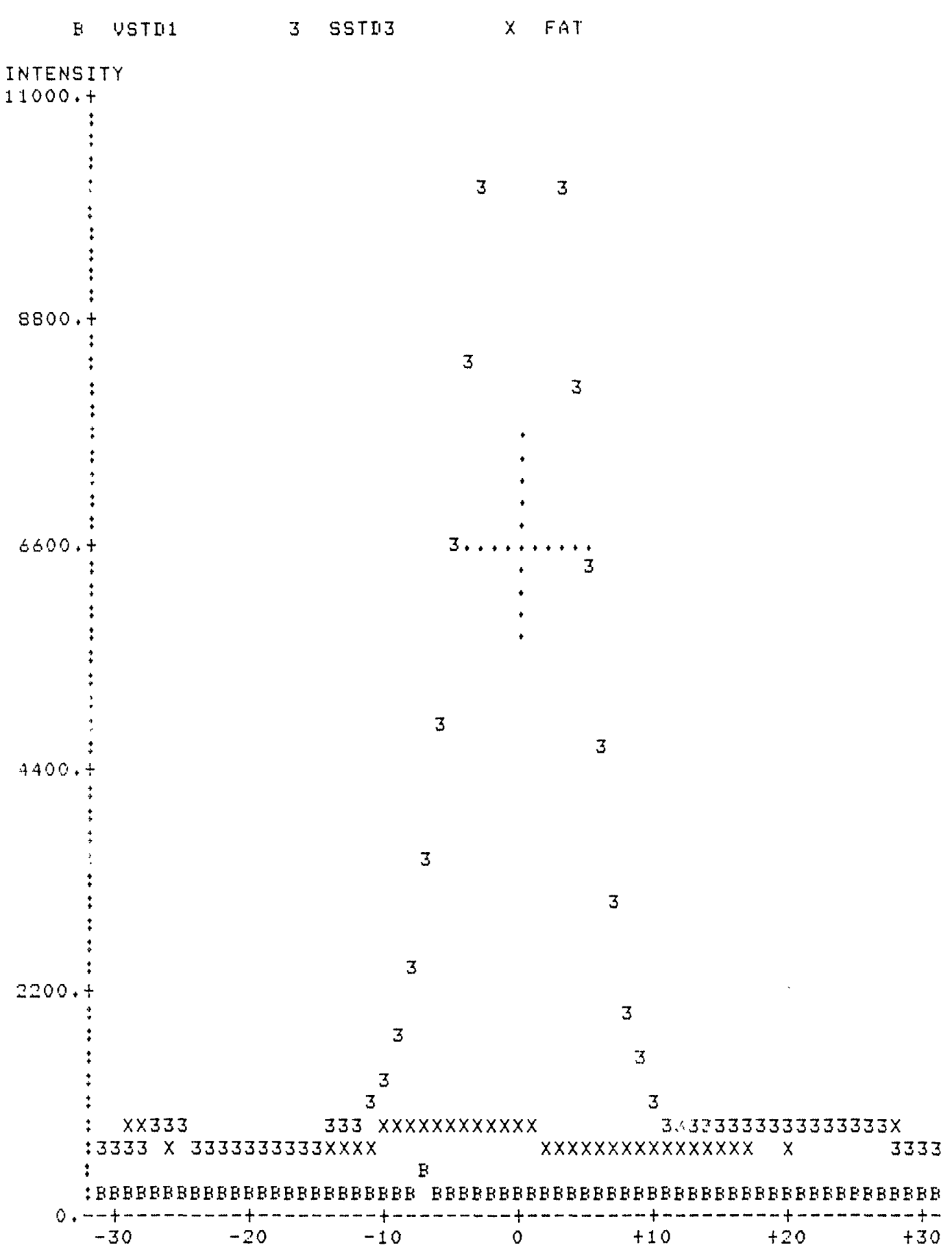
WAVELENGTH SCANS FOR PB AT 2203 ANGSTROMS



PEAK IS AT POSITION 1 HALF-WIDTH IS 9

WAVELENGTH SCANS FOR SB AT 2068 ANGSTROMS



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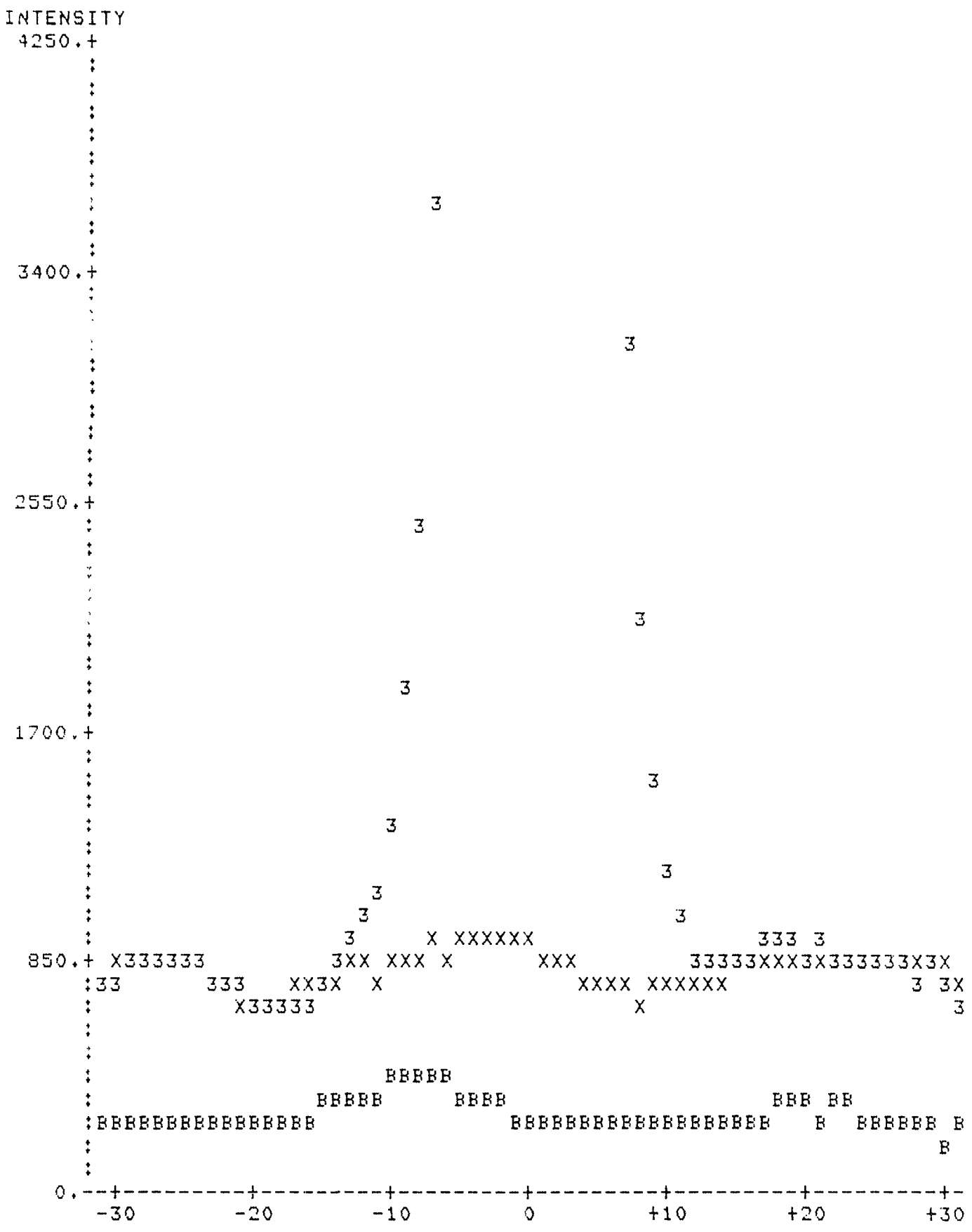
WAVELENGTH SCANS FOR SB AT 2068 ANGSTROMS



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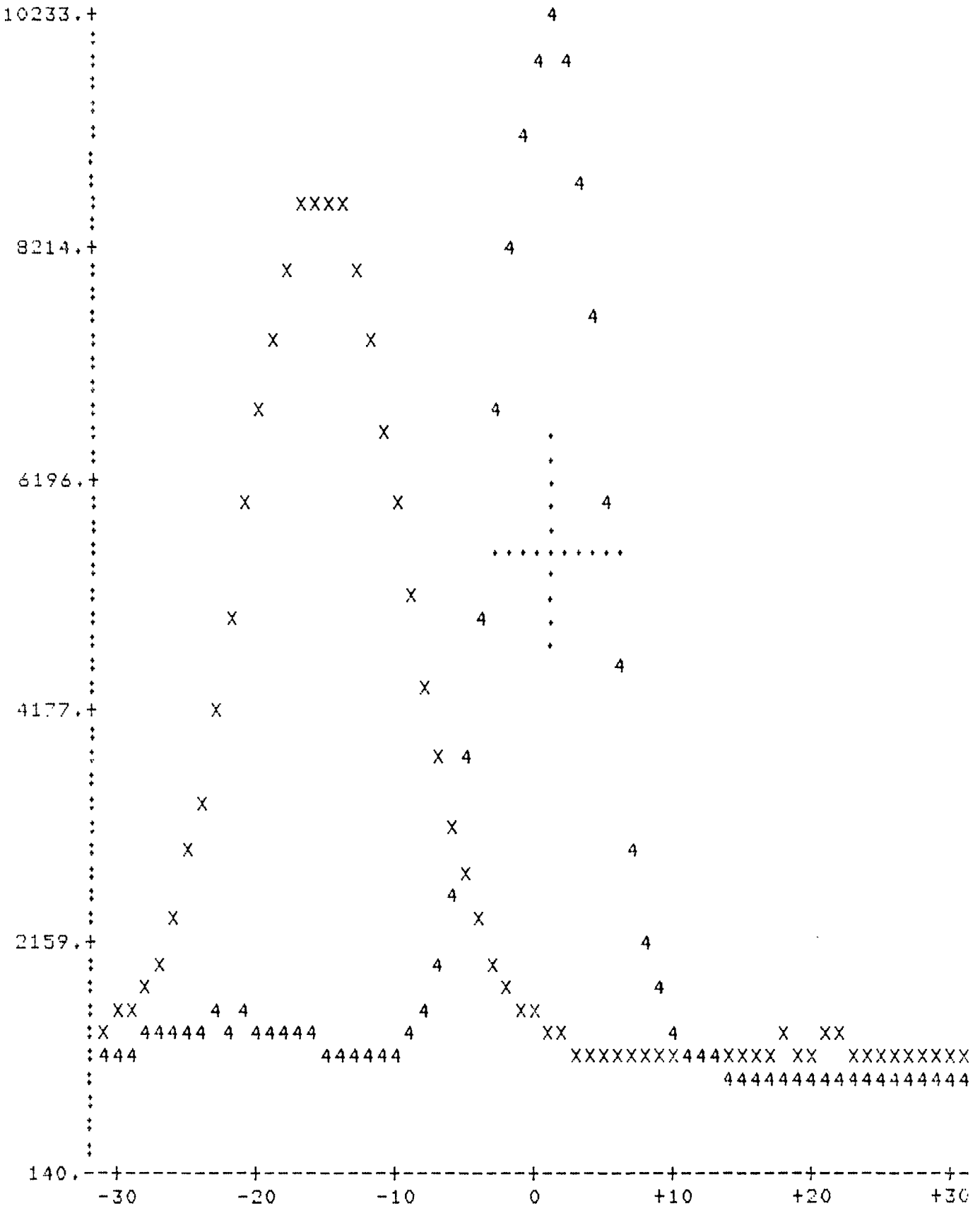
WAVELENGTH SCANS FOR SE AT 1960 ANGSTROMS

B VSTD1 4 SSTD4 X FAT



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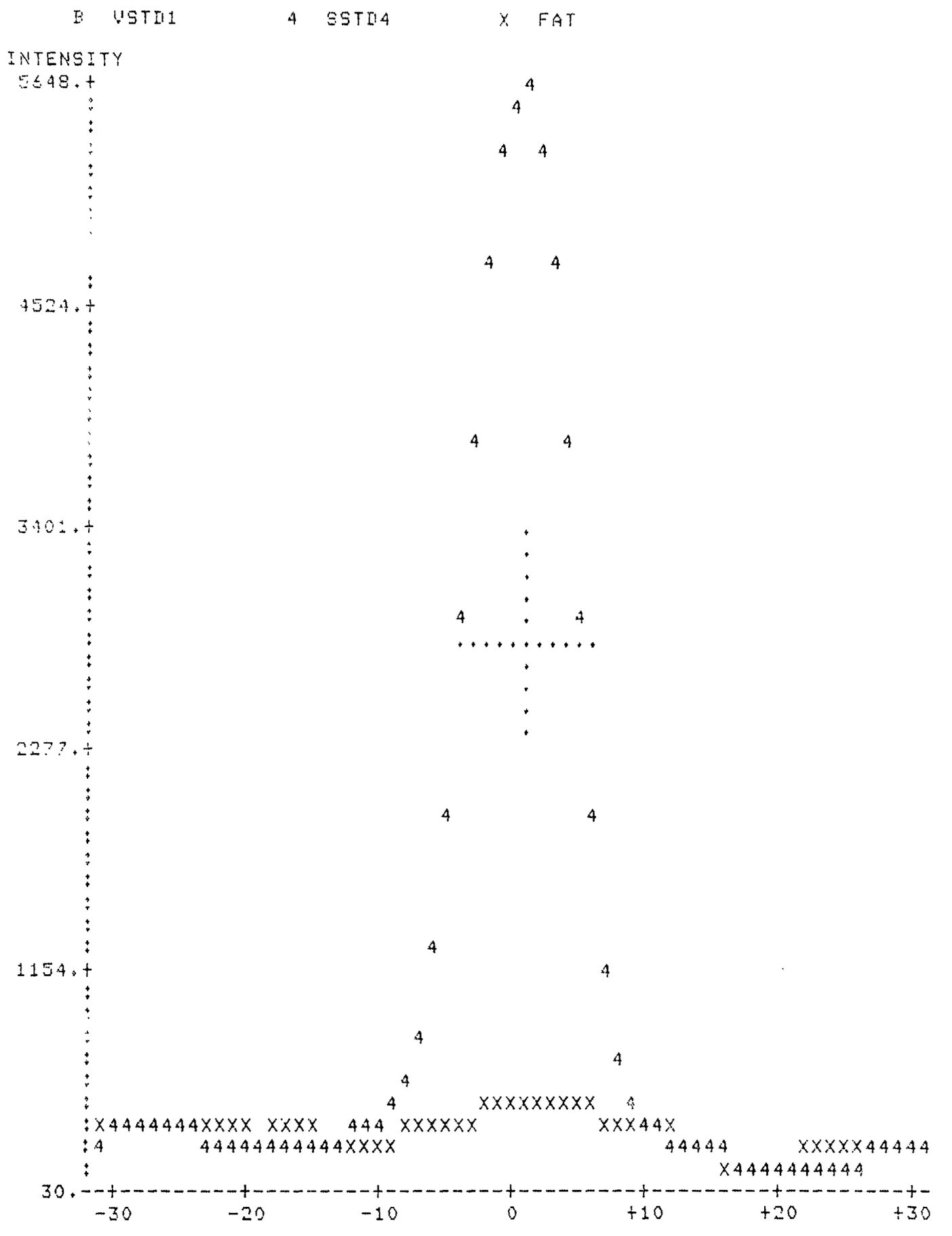
WAVELENGTH SCANS FOR [illegible] AT 1899 ANGSTROMS



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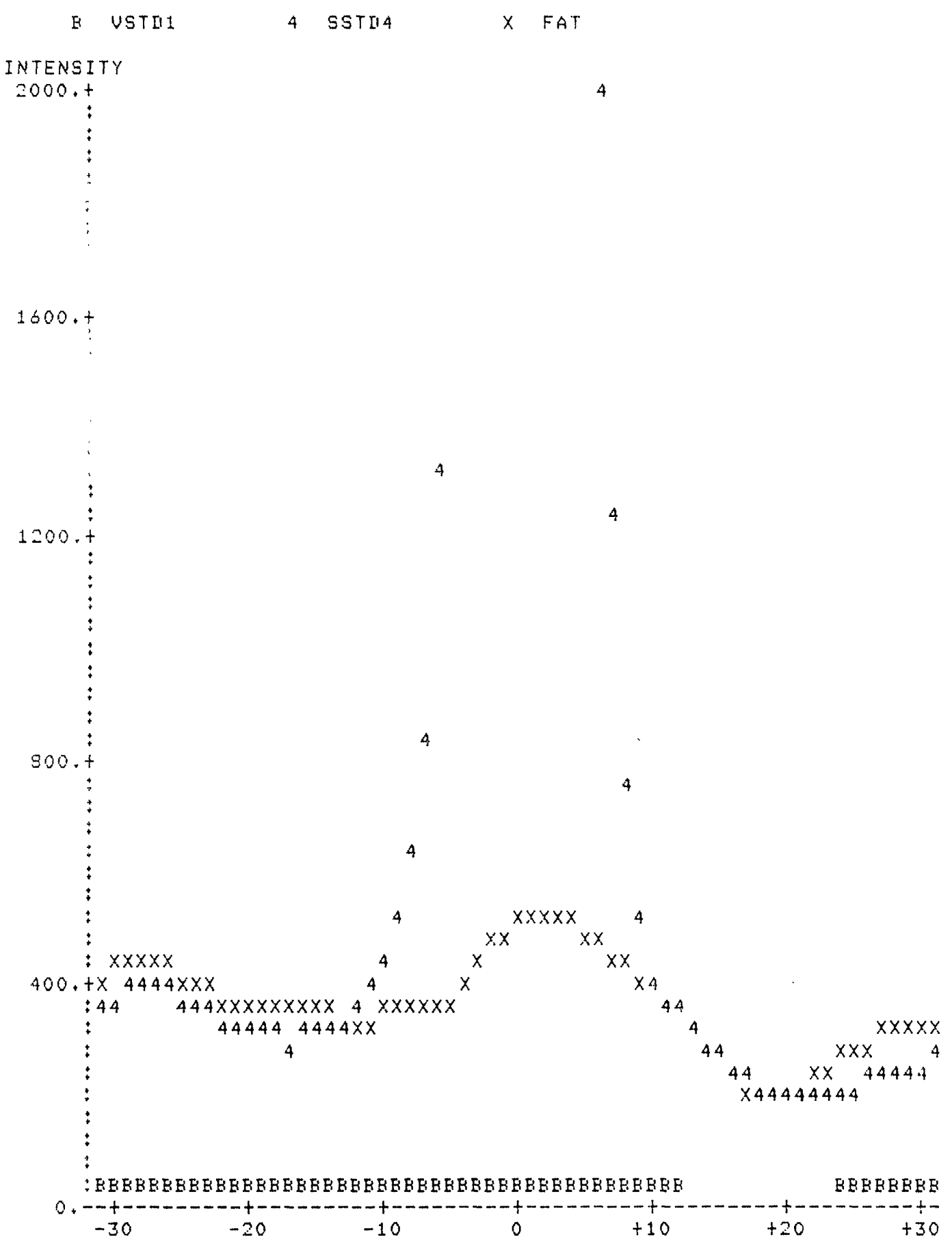
WAVELENGTH SCANS FOR SN AT 1899 ANGSTROMS



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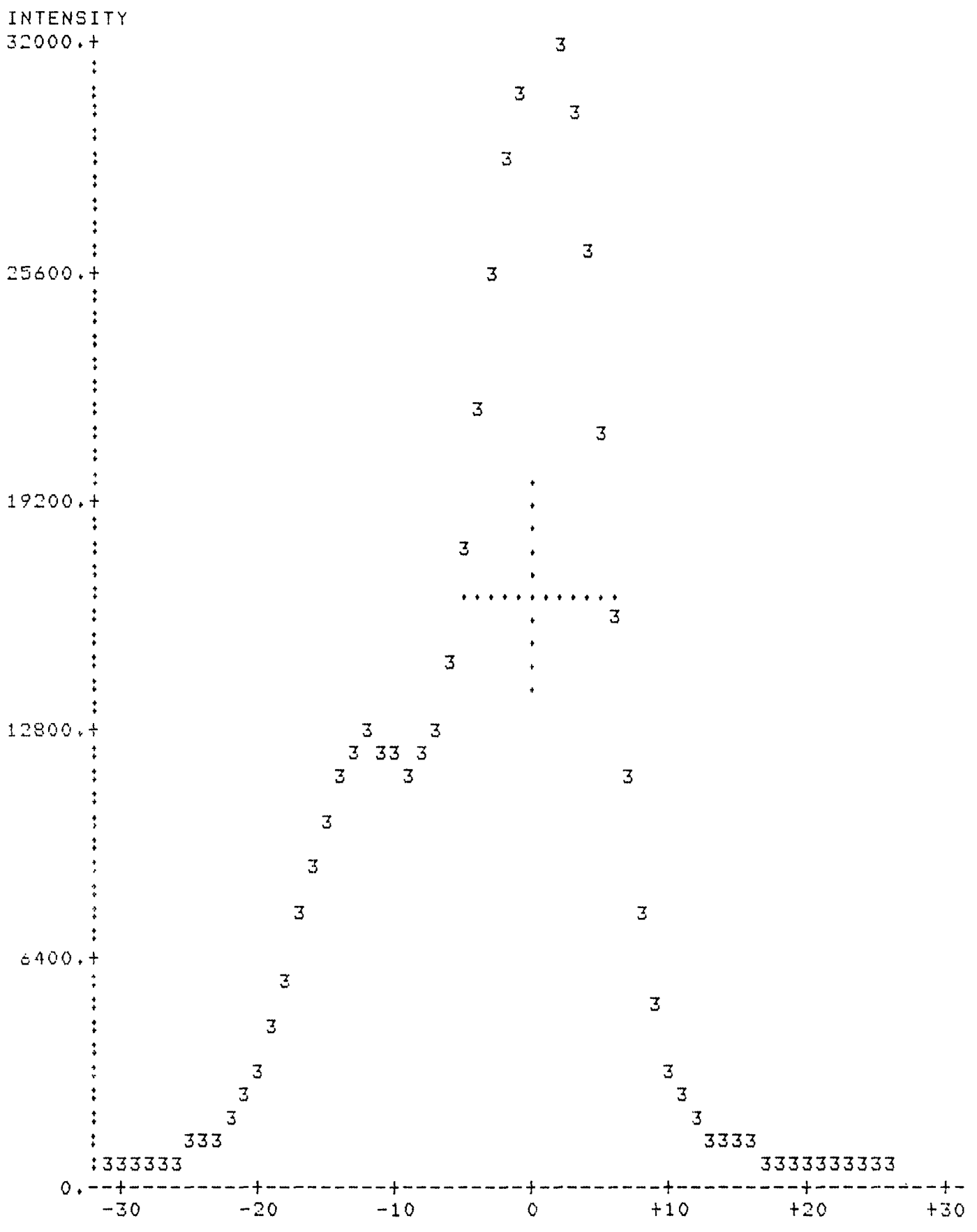

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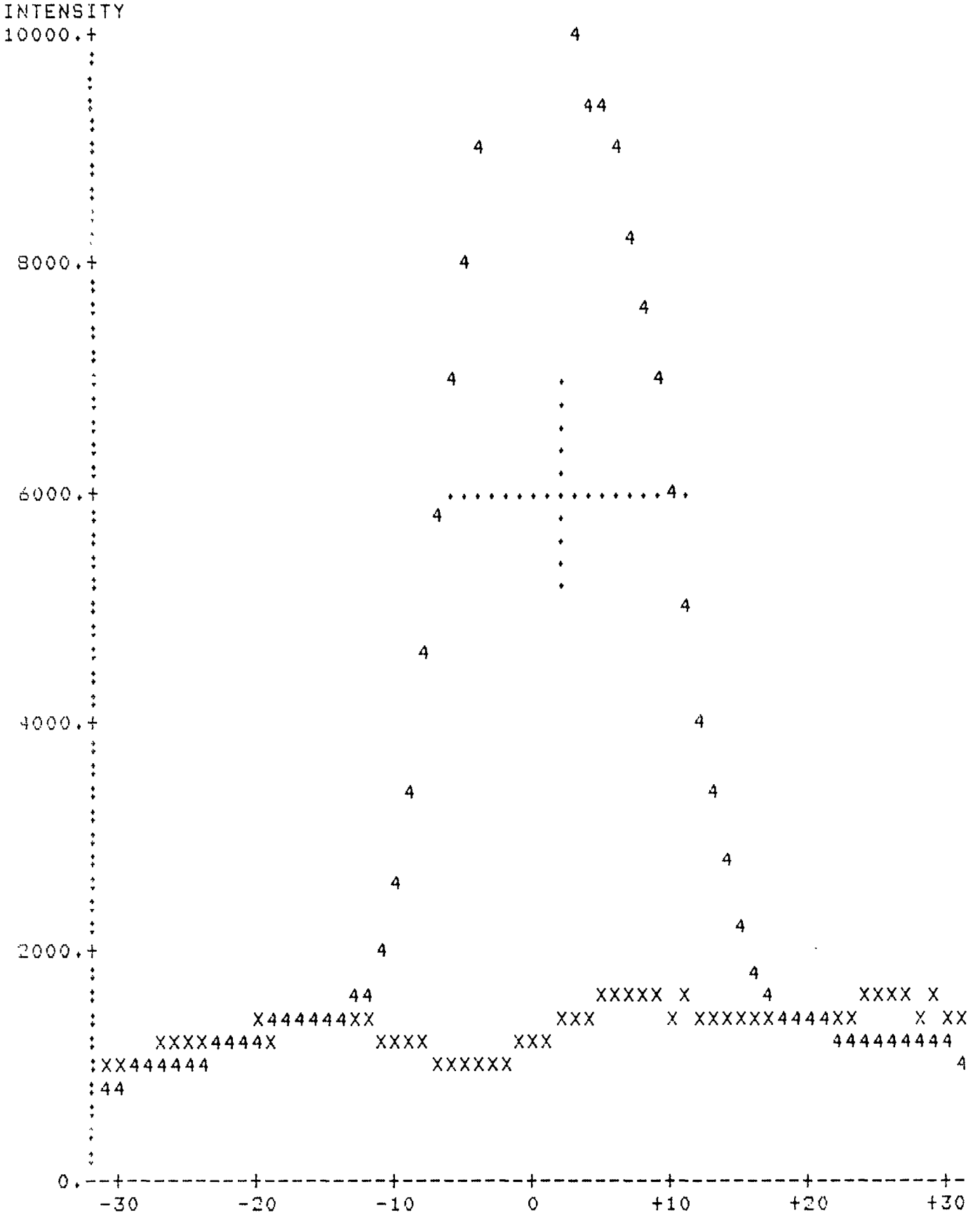
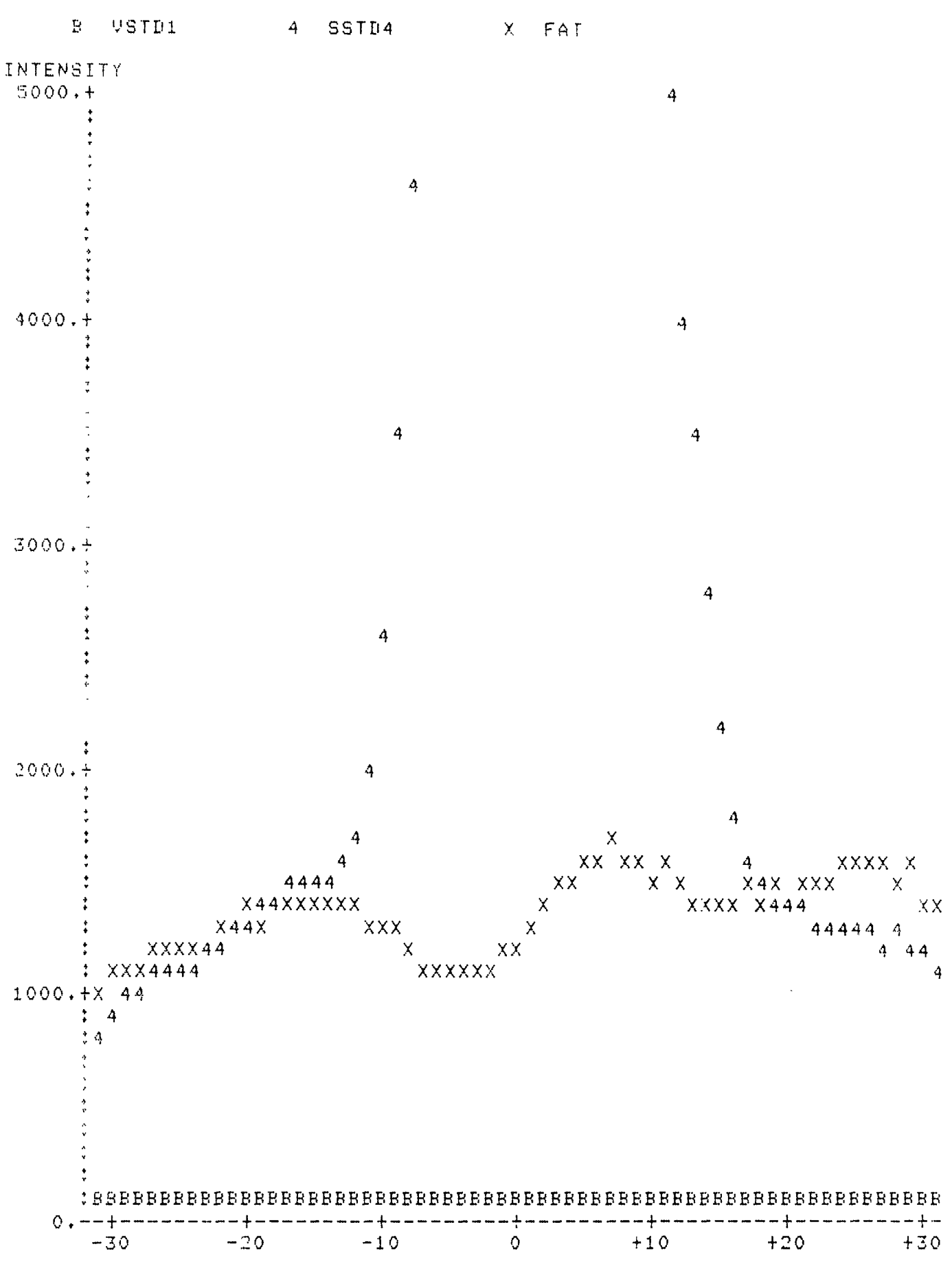
WAVELENGTH SCANS FOR TL AT 1908 ANGSTROMS

B VSTD1 4 SSTD4 X FAT



PEAK IS AT POSITION 2 HALF-WIDTH IS 18

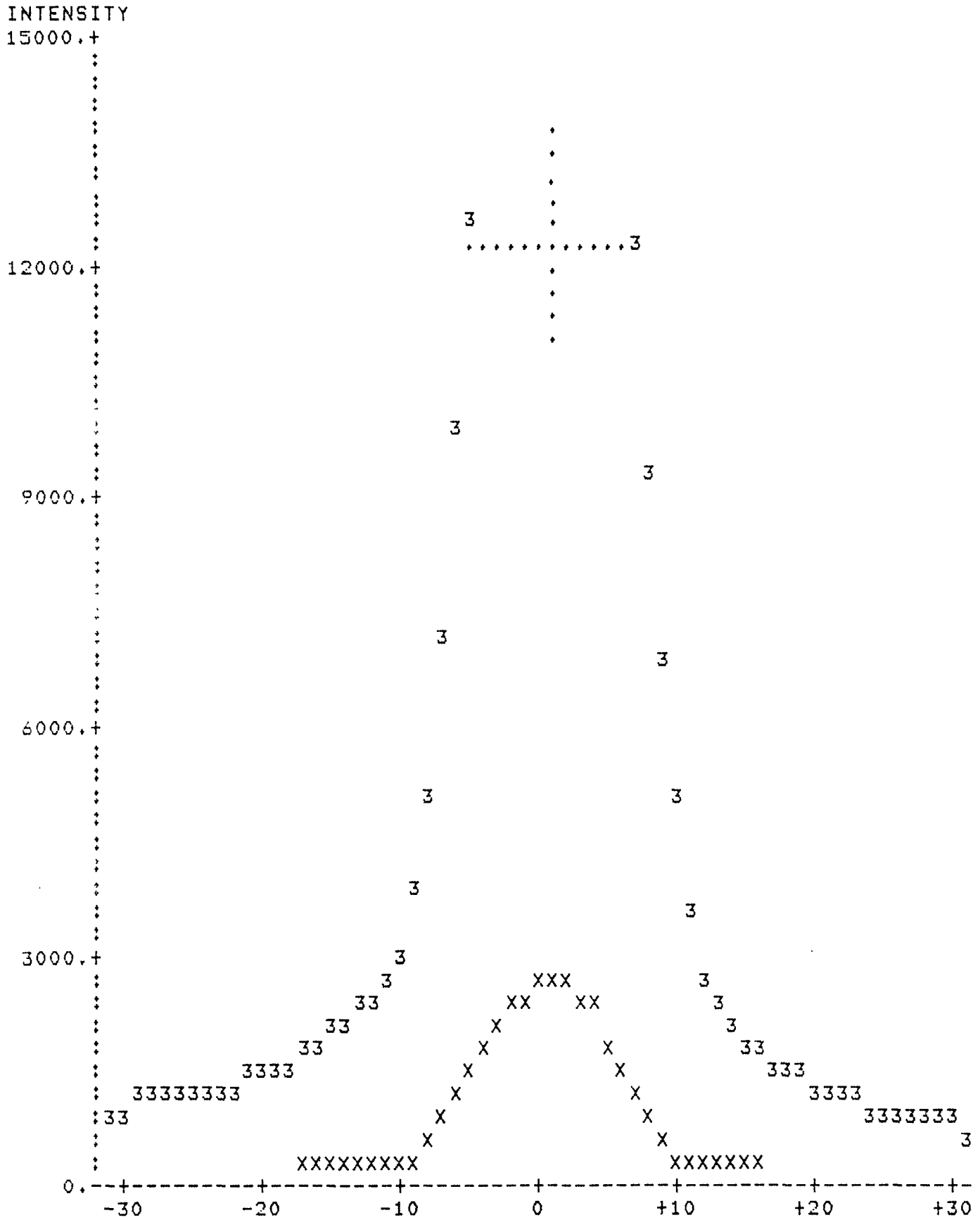
WAVELENGTH SCANS FOR TL AT 1908 ANGSTROMS



PEAK IS AT POSITION 2 HALF-WIDTH IS 18

WAVELENGTH SCANS FOR Y (INTERNAL STANDARD) AT 3710 ANGSTROMS

B VSTD1 3 SSTD3 X FAT



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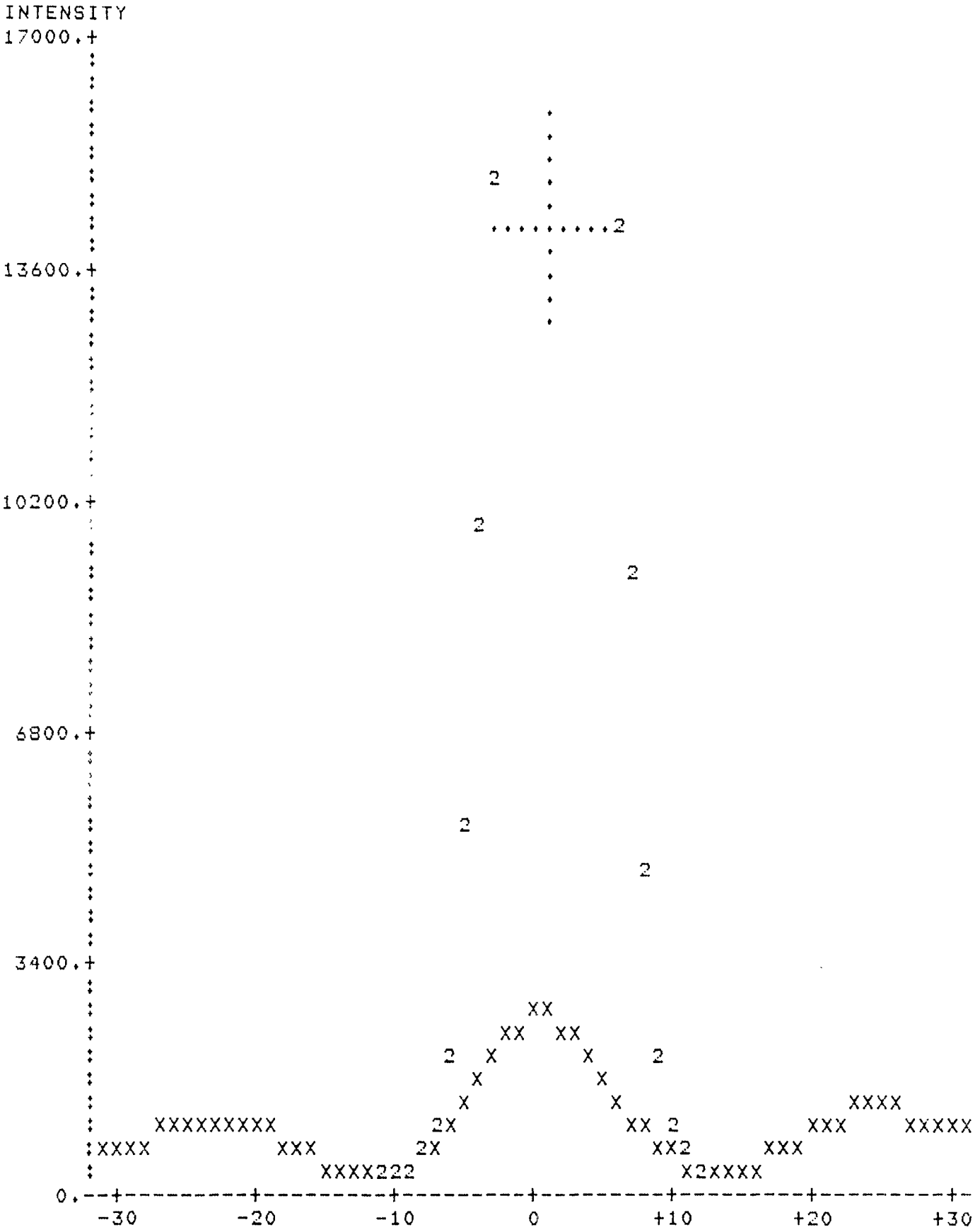
WAVELENGTH SCANS FOR ZN AT 2138 ANGSTROMS



PEAK IS AT POSITION 1 HALF-WIDTH IS 9

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(Please read Instructions on the reverse before completing)

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EPA-560/5-86-039		

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15. SUPPLEMENTARY NOTES
J. Remmers and P. Robinson, Work Assignment Managers
J. Breen and C. Stroup, Program Managers

16. ABSTRACT

The U.S. EPA's Office of Toxic Substances (OTS) maintains a unique capability for monitoring human exposure to potentially toxic substances through the National Human Adipose Tissue Survey (NHATS). NHATS is a statistically designed annual program to collect and analyze a nationwide sample of adipose tissue specimens for toxic compounds. The primary focus for NHATS has been to document trends in human exposure to environmentally persistent contaminants, specifically, organochlorine pesticides and polychlorinated biphenyls (PCBs). The NHATS specimens collected during fiscal year 1982 were designated for broad scan analysis. This broad scan analysis concept was applied to the determination of trace elements.

This report deals with the measurement of trace elements in selected adipose tissue specimen from the FY82 NHATS repository. The analyses of nine selected adipose tissue specimens from the FY82 NHATS repository were completed using two multielement techniques: inductively coupled plasma-atomic emission spectroscopy (ICP-AES) and neutron activation analysis (NAA). A total of 18 elements were detected using the two techniques and the estimated tissue levels are reported.

17. KEY WORDS AND DOCUMENT ANALYSIS

a. DESCRIPTORS	b. IDENTIFIERS/OPEN ENDED TERMS	c. COSATI Field/Group
Human Adipose Tissue Trace Elements Neutron Activation Analysis (NAA) Inductively-coupled plasma-atomic emission Spectroscopy (ICP-AES)	Analysis Determination	

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